The University of Nottingham

UNITED KINGDOM · CHINA · MALAYSIA

Department of Civil Engineering

Nottingham Transportation Engineering Centre (NTEC)

Meso-scale Rheological Characteristics of Foamed Bitumen Mixtures with High RAP Content

Diana B. Sánchez

Thesis submitted to the University of Nottingham for the degree of Doctor of Philosophy

Supervised by:

Prof. Gordon Airey, Dr. James Grenfell and Dr. Luis C. Neves

January 2018

ABSTRACT

The increasing concerns to reduce the production of harmful emissions, decrease energy consumption and preserve the natural resources have motivated the asphalt industry to lower the production temperatures of asphalt mixtures and incorporate alternative materials. One approach is to use Foamed Bitumen Mixtures (FBM) in combination with Reclaimed Asphalt Pavement (RAP) material. FBMs are composed of a combination of foamed bitumen and mineral aggregates, where unlike conventional HMA where the bitumen is in a liquid state during mixing, the bitumen present in these mixtures is in a foamed state during the mixture's production. Foamed bitumen production has been mainly based on the techniques developed more than 50 years ago, where the bitumen is heated at high temperatures and then foamed by combining it with cold water, a process that is also known as *mechanical foaming*. In its foamed state, the bitumen has lower viscosity and expanded volume and can, therefore, be mixed with cold (and often damp) aggregates. These two conditions allow bitumen in its foamed state to coat the aggregates at lower temperatures in comparison to those required in regular HMA materials. More recently, new foaming technologies have been developed as part of Warm Mix Asphalt (WMA) technologies. WMA are asphalt mixtures that are produced at reduced temperatures (i.e. mixing temperatures around 120°C) compared to traditional HMA materials, obtaining mainly environmental and economic benefits. The possibility of reducing the mixing temperature for the asphalt mixture by using these types of foaming technologies is achieved through the use of a series of products and/or processes that act on the bitumen through different mechanisms to provide the adequate workability and coatability conditions when combined with the aggregates at these reduced temperatures.

On the other hand, RAP materials are essentially aged asphalt pavements that have reached the end of their service lives and are reclaimed to be used as part of the construction or rehabilitation of pavement structures. The incorporation of this material in Hot Mix Asphalt (HMA) reduces the amount of raw materials added to the final mixtures, which contributes to the conservation of natural resources. Thus, the use of foaming technologies together with the great potential shown by RAP materials to be successfully recycled in asphalt mixtures, are encouraging the use of these materials in road infrastructure projects, thereby requiring the study of their fundamental properties and providing an indication of their expected field performance. This evaluation is particularly important taking into account that a main concern related to the use of lower production temperatures of the mixtures is if the properties of the material are still comparable to those of the traditional HMA. Furthermore, in the case of RAP materials it is still unclear what role the old RAP bitumen plays in the presence of virgin materials, particularly because the blend of RAP-virgin bitumen has a great influence on the final

performance of the produced mixtures. Thus, the use of foamed bitumen in mixtures that also include RAP constitutes an important subject of study.

This thesis is concerned with the use of the Dynamic Mechanical Analysis (DMA) testing technique as part of a comprehensive experimental methodology to evaluate the rheological response of foamed bitumen mixtures produced by the traditional mechanical foaming technique, and by the incorporation of a foaming additive based on zeolite minerals commonly used in warm mix technologies, and their behaviour in the production of mixtures containing high RAP content (i.e. 50% by total weight). The relevance and suitability of the testing methodology was evaluated at the meso-scale by testing specimens representing the fine aggregate matrix (FAM) or asphalt mortar existing in the full mixtures which has a relatively more uniform (or less heterogeneous) internal structure and it has been reported to accurately represent the mechanical behaviour of the whole asphalt mixture (Masad et al. 2006). In this study, FAM is defined as a combination of bitumen, with mineral aggregates of particles size smaller than 1 mm. Initially, the rheological characteristics of the FBMs produced with both foaming technologies were studied. This was followed by the evaluation of the rheological response of these mixtures with RAP material. In all cases, this evaluation included the effect of production temperature for half-warm (i.e. mixing temperatures of 90^{o}C), warm (i.e. mixing temperatures of 120^{o}C), and hot processes (i.e. mixing temperatures of 160^{o}C).

Experimental results of applying DMA testing on FAM FBMs identified the characteristics of foamed bitumen mixtures manufactured with the two different foaming technologies. It was found that the foaming technology and production temperature have different impacts on the produced mixtures. In the case of the mechanical foamed mixtures, the rheological characteristics of these mixtures were found to be strongly dependant on the production process and the mixing temperature of the materials. Conversely, changes in the production temperature do not have a considerable effect on the rheological characteristics of the zeolite containing mixtures. When 50% RAP material was incorporated the properties of the mixtures were found to be influenced by the level of blending between RAP-virgin binders, which was found to be mainly a function of the foaming technology and the final temperature of the mixtures.

The present work was also intended to evaluate how the fundamental rheological properties of these FAM FBMs with RAP material are affected by several environmental conditions, such as the combined effects of water and oxidative ageing. This was achieved by applying two conditioning procedures including the BS EN 12697-12:2008 with further conditioning in a 60^{o}C water bath at different times, and the Saturating Ageing Tensile Stiffness (SATS) protocols. It was found that in the presence of moisture and high temperature environments, the age-hardening related effects dominated the response of the FAM materials. These effects are believed to be linked to the fact that the FAM

type mixtures contain more bitumen and smaller size voids compared to the full asphalt mixtures, which could impact the rate at which moisture can reach the inner portion of the testing specimens, whereas high temperatures certainly lead to some ageing and stiffening of the materials.

ACKNOWLEDGEMENTS

I would like to extend my sincere gratitude to Professor Gordon Airey, my main supervisor, for his continuous support, guidance and excellent supervision throughout the development of this research. Despite his heavy workload, he was always willing to advise, share ideas and provide interesting discussions which kept me motivated. It has been a pleasure to be part of his research group.

I am also grateful to Dr. James Grenfell and Dr. Luis C. Neves for their technical support and great advice. My gratitude also goes to Dr. Silvia Caro, Associate professor at Universidad de Los Andes (Bogotá, Colombia) for her technical and emotional support and her relevant contributions to this research, which added quality value.

I would also like to acknowledge Richard Blakemore, Lawrence Pont, Jon Watson, Martyn Barrett, Antony Beska, Matt and Laura, the team of technicians in the laboratory, for their hard efforts to prepare specimens and set-up experiments, and also for generating a nice environment to work in.

I would also like to thank all my fellow researchers and all the academics at NTEC for making this experience unique and meaningful. I especially want to acknowledge Ana Jiménez del Barco and Breixo Gómez for their invaluable friendship, and for sharing with me so many unforgettable academic and personal experiences.

My deepest gratitude goes also to my beloved parents for their permanent encouragement, support, and for been my source of strength. Many thanks to my dear sister Valentina, my brother and my eldest sister for their unconditional support.

I appreciate the financial support provided by Colciencias throughout the program 617 for doctoral studies abroad, and the partial funding provided by NTEC that made this work possible.

DECLARATION

The research described in this thesis was conducted at The University of Nottingham, Nottingham Transportation Engineering Centre between April 2014 and April 2017. I declare that the work is my own and has not been submitted for a degree of another university.

Diana B. Sánchez
University of Nottingham
January 2018

Table of Contents

List of Figures

List of Tables

LIST OF PUBLICATIONS

- Sánchez D.B, Grenfell J, Airey G, and Caro S (2017). Evaluation of the degradation of fine asphalt-aggregate mixtures containing high reclaimed asphalt pavement contents. *Road Materials and Pavement Design*. April 2017.
  http://dx.doi.org/10.1080/14680629.2017.1304250

- Sánchez D.B, Airey G, Grenfell J, and Caro S (2017). Moisture effects on the properties of RAP-foamed bitumen mixtures. *Tenth International Conference on the Bearing Capacity of Roads, Railways and Airfields (BCRRA 2017)*, Athens, Greece, 28 – 30 June 201

Chapter 1 : Introduction

1.1 Background

A road is an essential component of the transport infrastructure, so it plays a fundamental role in the development of a society. Roads are composed of a multi-layered system that consists of a sequence of selected and processed materials that are placed on the natural ground or subgrade. They are expected to function properly by providing a smooth traveling surface for the traffic under various conditions of the environment. The ultimate purpose of the whole structure is to distribute the stresses produced by the traffic loads to the subgrade, to ensure that the bearing capacity of this layer is not exceeded during the design life of the road (Mallick and El-Korchi, 2013).

Road structures can be broadly classified into flexible (or asphalt) and rigid (or concrete) pavements. Flexible pavements are the most common type used for road construction. In the UK, around 90% of the roads are constructed with flexible pavements (Thom, 2014). Hot Mix Asphalt (HMA) is the main material used for the construction of the surface and structural layers of the flexible pavement and is mainly composed of a combination of mineral aggregates (i.e. crushed rocks with different sizes and proportions) and bitumen, the latter being a by-product of the petroleum distillation process.

Traditionally, these mixtures are produced using hot processes by heating up the bitumen and aggregates to very high temperatures (i.e. 135 - 160^{o}C) (Kuennen, 2004, Newcomb, 2005). Bitumen heating is required to reduce its viscosity and enable coating of the aggregates and ensure mixture workability. Heating of the aggregate is required to remove the moisture present on the aggregates to help the bitumen adhere more easily to its surface. However, at such elevated temperatures, great amounts of harmful emissions and fumes are generated, contributing to environmental contamination. At the same time, due to the continuous growth in the transportation area, and the consequent demand to construct new roads or rehabilitate the existing ones, there is a constant need of the constituent materials of these mixtures (i.e. bitumen and aggregates) resulting in scarcity of natural resources. In the UK, for example, there are approximately 14,000 km of trunk road and motorway between 5 and 10% of which have to be resurfaced each year. In the USA, this figure is estimated to be about six times higher (Thom, 2014). Therefore, sustainability and environmental related reasons justify the need to use lower production temperatures and incorporate alternative materials.

One approach in this direction is to incorporate large quantities of Reclaimed Asphalt Pavement (RAP) material into surface course applications in combination with Foamed Bitumen Mixtures (FBM). Foamed bitumen mixtures have been traditionally produced using a mechanical foaming technique in which hot liquid bitumen is combined with a stream of cold water to generate foam, for cold bitumen mixtures applications (i.e. aggregates at ambient temperature) (Csanyi, 1957), with some extensions for half-warm processes, by combining the foamed bitumen with aggregates heated up to 100^{o}C (Jenkins et al., 2000). In addition, this material has been used as part of various stabilization processes, which also include the use of RAP. More recently, new foaming techniques have been developed as part of WMA materials. WMA are asphalt mixtures that are produced and compacted at lower temperatures than those typically used in the production of traditional HMA. The possibility of reducing the mixing temperature for the asphalt mixture by using foaming technologies is achieved through the use of a series of products and/or processes that act on the bitumen through different mechanisms to provide the adequate workability and coatability conditions when combined with the aggregates. There are several types of WMA foaming technologies, which include the use of zeolite additives, the incorporation of wet fine aggregates (Low Energy Asphalt, LEA), or a two-component bitumen system (WAM-Foam) that includes mechanical foaming technique (D'Angelo et al., 2008, Perkins, 2009). It has been shown that energy consumption of WMA production can be between 60 to 80% lower than HMA production, depending on the production technology (Hurley and Prowell (2005).

On the other hand, RAP material, which is obtained from milling existing distressed bituminous surfacing in pavement construction and rehabilitation works, has being regularly used in many countries to conserve natural resources. RAP often contains high-quality, well-graded aggregates that are coated with bitumen, the main constituent materials of asphalt mixtures, and therefore are reused for sustainability and environmental purposes mainly. With the incorporation of this material, the amount of new materials added to the final mixtures are reduced thus, constituting a sustainable and environmentally friendly technology (Copeland, 2011). In Europe, 300 million tonnes of hot mix asphalt are produced annually, and it is estimated that over 50 million tonnes of RAP are produced each year with, in some instances, over 70% of it being reused for road surfaces. According to the European Asphalt Pavement Association (EAPA), the goal is to achieve 100% in the upcoming years. All in all, the use of RAP material and foaming technologies fulfil several of the requirements set by environmental agencies to preserve natural resources and reduce environmental contamination, thus the combination of these materials constitute an attractive alternative recycled material for the asphalt industry.

1.2 Problem statement

As with any new technology and combination of materials proposed, it is important to evaluate their fundamental characteristics and provide an indication of their expected field performance. In the case of RAP, during the construction and service life of the road from which it is obtained, the bitumen is exposed to the passing traffic loads, and the existing weather conditions in the area of the project, resulting in significant changes in its physical and rheological properties. Therefore, when RAP material is incorporated into a new mixture, it is paramount to suitable account for the impact of the old material in the design of the new RAP asphalt mixtures. In addition, the production process and mixing temperatures of the asphalt mixtures also play a significant role in their final properties. In the case of WMA technologies, it is expected that the reduction in the production temperature does not affect negatively the performance of the final mixtures, so they could be considered an effective replacement of conventional HMA.

The overall goal of this research study is to evaluate the rheological characteristics of asphalt mixtures manufactured with two different foaming technologies (i.e. a mechanical foaming technique and the incorporation of zeolite foaming additives), and their behaviour when high RAP content (i.e. 50%) is incorporated. In all cases, this evaluation also includes the effects of production temperature for half-warm, warm and hot applications.

Initially, the study investigates the rheological response of the asphalt mixtures produced with both foaming technologies, with no RAP material added, in order to understand the effects of the foaming technology and production temperature on the final response of these materials. Then, half of the components of these mixtures are replaced by RAP in order to evaluate the effect of the incorporation of this material in the final properties of all the mixtures. As part of this aim, the level of blending between the old bitumen from RAP and the virgin bitumen is also evaluated.

Furthermore, this study also evaluates how these rheological properties are affected by several environmental conditions such as the presence of moisture and oxidative ageing. Hence, this research is expected to provide new and unique information to understand the advantages and limitations of these materials, and provide an insight of their expected durability in the field.

1.3 Research objectives

The overall objectives of this study are:

- Evaluate the difference in the viscoelastic behaviour of foamed bitumen mixtures produced by means of a mechanical process and by the addition of zeolite foaming additives at various mixing temperatures (half-warm, warm and hot applications).

- Analyse the rheological viscoelastic behaviour of asphalt mixtures produced with the same two foaming technologies with 50% of RAP material, including also various mixing temperatures for half-warm, warm and hot applications. As part of this objective, the level of blending between the RAP and virgin bitumen present in these mixtures was also assessed.

- Evaluate the effects of water infiltration and oxidative ageing on the rheological response of the RAP-FBMs. As part of this objective two conditioning processes were evaluated. The first one incorporates the water sensitivity test commonly used in the UK - British Standard BS EN 12697-12:2008- including further conditioning in a water bath at 60ºC for different periods of time, and the second one consists of the application of the Saturation Ageing Tensile Stiffness (SATS), which is considered a more severe conditioning protocol to evaluate these effects.

1.4 Research methodology

With the objective of properly understanding the rheological characteristics of the foamed bitumen mixtures manufactured with both foaming technologies, the initial task in this work consisted of evaluating these set of mixtures individually. Then, 50% of the components of these mixtures were replaced by RAP in order to understand the effect this material has on their rheological response. In all cases, the evaluation includes the effect of production temperature on the rheological response of the produced mixtures. For the second set of mixtures – containing RAP material – the evaluation included the level of blending between the RAP-virgin binders with production temperature.

The evaluations previously described were conducted by testing specimens representing the fine matrix of the full mixtures. In other words, the study was conducted at the ‘meso’-scale. Specifically, for this study Fine Aggregate Matrix (FAM) materials are composed by a combination of fine particles below 1mm, filler (passing 0.063mm), bitumen and air voids. The advanced characterisation

of the viscoelastic properties of the evaluated mixtures was conducted by applying the Dynamic Mechanical Analysis (DMA) testing technique on the FAM materials, performing frequency and temperature sweeps tests. DMA is a powerful tool that has been successfully used to characterise the viscoelastic properties (i.e. linear and non-linear) and the fatigue deterioration process of the FAM materials (Masad et al., 2006a, Masad et al., 2008, Caro et al., 2008a, Caro et al., 2012). In addition, this method has several advantages as it uses a small amount of material compared to testing full asphalt mixtures, it requires reasonable testing time and since the technique uses a regular rheometer, it does not involve the development of new advanced equipment or the adaptation of existent ones.

The durability of the foamed bitumen mixtures containing RAP material was evaluated following two conditioning protocols. The first one consisted of following the moisture conditioning protocol suggested by BS EN 12697-12:2008 but after including further conditioning in a water bath at 60ºC for 6, 15 and 30 days. This conditioning protocol allowed to track the rheological changes occurring in each type of mixture due to the presence of moisture and oxidative ageing with time.

The second protocol consisted of the application of the Saturating Ageing Tensile Stiffness (SATS) test on the FAM-RAP-FBMs. This conditioning protocol, which also includes the combined effects of moisture and oxidative ageing, constitutes a more severe approach that was used to further understand the impact of these environmental conditions on these types of materials. As part of this evaluation, the effects of the SATS conditioning protocol on the rheological properties of the bitumen present in the RAP-FBMs was also studied. The binders were recovered by distillation following the BS EN 12697-4:2005 standard and the viscoelastic characteristics were measured by conducting frequency and temperature sweeps test by means of the Dynamic Shear Rheometer (DSR). Since the asphalt mixture's performance and properties are highly influenced by the characteristics and rheological behaviour of their bitumen, this bitumen characterisation will provide fundamental information to interpret and justify the corresponding mixture's response. In addition, this information will provide an insight of the level of blending occurring between the RAP and virgin bitumen.

1.5 Structure of the study

This document consists of eight chapters. The present chapter (Chapter 1) demonstrates the significance of the topic, describes the research objectives, and provides the general outline for the thesis.

Chapter 2 contains a theoretical background and previous studies on the main materials used in this study. It includes a description of both foaming technologies evaluated, the challenges of

incorporating RAP material in HMA mixtures, the bitumen blend design to account for the RAP bitumen in the final mixture, the viscoelastic characterisation of asphalt materials, and the effects of environmental factors on the rheological response of the materials.

Chapter 3 describes the design of the FAM mixtures and the procedure to manufacture all the FAM mixtures evaluated in this work. In addition, this chapter presents the DMA experimental set-up for testing the FAM materials and the DSR for binders.

Chapter 4 focuses on the quantification of the rheological characteristics of fine foamed bitumen mixtures produced by means of a mechanical process and by the addition of zeolite foaming additives. This quantification includes the effect of production temperature in the final properties of the mixtures. Furthermore, the last part of this chapter includes the rheological characteristics of the recovered binders from these mixtures.

In Chapter 5, 50% of the components of the mixtures produced in Chapter 4 were changed to incorporate RAP material, and investigate the effects in the rheological characteristics of the final mixtures. As in Chapter 4, these mixtures were evaluated at the meso-scale for half-warm, warm and hot processes. This chapter includes the evaluation of the level of blending between the old and new materials.

In Chapter 6, following the results obtained in Chapter 5, the most representative fine mixtures for paving applications were selected and the influence of an environmental conditioning process that incorporates the effects of water infiltration and oxidative ageing on the rheology of the mixtures was evaluated.

In Chapter 7, the impact of a more severe combined ageing-moisture sensitivity laboratory conditioning protocol such as the Saturation Ageing Tensile Stiffness (SATS) on the rheological response of the RAP-FBMs was evaluated. The last part of this chapter includes the rheological changes occurring in the bitumen present in the evaluated mixtures, after the SATS conditioning.

Finally, Chapter 8 summarizes the main conclusions obtained from this thesis and proposes some areas of future work.

Chapter 2 : Theoretical background and previous studies

This chapter presents the most relevant background information for the understanding of the topics treated along this research work. It starts with a general overview about the pavement structure in flexible pavements, followed by the main classification of asphalt mixtures according to their production temperature range, and the potential benefits of reducing the production temperature of asphalt mixtures as a replacement for HMA. The following two sections focus on describing the main materials used in this work. The first section focuses on Warm Mix Asphalt (WMA) materials, where the three main groups of technologies available to produce these type of mixtures are introduced, and the following parts of this section are emphasised solely on foaming technologies as they constitute one of the main focus of this study. The second main section concentrates on the RAP materials, describing the main challenges of the incorporation of this material into HMA and the bitumen blending model mostly used in Europe, to account for the properties of the RAP bitumen in the final mixture design.

After the main materials used in this study are described, the different scales for evaluation of bituminous materials are presented, including the FAM materials. This section also contains previous research efforts performed using this type of material. Next, the main theoretical information related to the rheological characterisation of these materials, including fundamental viscoelastic concepts that are employed in this study, along with the rheological models applied for the analysis of the rheological data, are presented. Lastly, two of the most commonly used damage factors that affect the durability of asphalt mixtures, named moisture damage, and age hardening are described, including the tests producers available to evaluate these effects on asphalt mixtures.

2.1 Background

A flexible road structure consist of a number of layers of different materials, as illustrated in Figure 2.1. In structural terms, the aim of a road pavement is to support the loads induced by traffic and to distribute these loads in such a way that the transmitted stresses do not exceed the capacity of the subgrade (Airey, 2010, Mallick and El-Korchi, 2013)

Figure 2.1: Typical pavement structure of a flexible road

The main function of these layers are described below (Mallick and El-Korchi, 2013, Thom, 2014):

- Surface course: it is the layer that is directly in contact with the vehicles, and normally contains the highest quality materials. It has to withstand the maximum stress produced by the traffic loads and bear the changing conditions of the environment. Also, this layer provides a dense surface to restraint water infiltration into the underlying layers, and its surface properties are designed to achieve adequate skid resistance.
- Binder course: this layer acts as an intermediate regulating layer, which is very similar in composition to the previous layer, but it may comprise larger size aggregates.
- Base course: it is the layer located in the middle of the structure and its main function is to provide an overall stiffness to the pavement structure such that it does not deflect too much under load.
- Subbase course: the function of this layer is similar to that of the previous layer, except that it could be of lower quality materials since the stress provided by the traffic loads is lower at this level.

- Capping: is an improvement layer for the natural soil, which generally consist of mixing the natural soil with lime, cement or a hydraulic binder.
- Subgrade: this layer corresponds to the natural soil. It serves as foundation to the whole pavement structure, and should be of such quality to resist excessive deflection under load. For this reason its properties are usually enhanced with the capping layer.

The nature of the materials vary according to their position and function in the structure. Thus the first three layers (i.e. surface course, binder course and base) shown in Figure 2.1 may contain asphalt materials but the properties required of the surface course, which is in contact with the wheels of the vehicles, are different from those required just below the surface in the binder course and base (Airey, 2010). The asphalt mixtures evaluated in this research are studied for their application in the surface course of flexible roads.

Asphalt mixtures can be classified by the production temperature in four categories (D'Angelo et al., 2008):

- Cold mix asphalt (0-30ºC)
- Half-warm mix asphalt (65 – 100ºC)
- Warm mix asphalt (100 – 140ºC)
- Hot mix asphalt (above 140ºC)

Typically, HMA is produced at 140 – 160ºC. WMA is produced at temperatures that are 20 – 30ºC below HMA, to temperatures slightly above 100ºC. Warm mix asphalt mixes are separated from half-warm asphalt mixtures by the resulting mix temperature. If the resulting temperature of the mix at the plant is less than the boiling point of water (i.e. 100ºC), the mix is considered half-warm mix. Finally, cold mixes are produced near ambient temperature (D'Angelo et al. 2008).

2.2 Warm mix asphalt

The temperature reduction to produce these type of mixtures, is achieved by the use of various products and/or processes, to enhance workability of the mixture, increase mixtures' lubricity or reduction in the viscosity of the bitumen, whilst providing adequate aggregate coating at these substantially reduced production temperatures (Bonaquist 2011). In all cases, the goal to produce these mixtures is to generate environmental, energetic and constructive benefits. Some reported benefits of WMA include (Bonaquist 2011):

- Reduced plant emissions
- Reduced fuel usage

- Reduced worker exposure to fumes
- Paving benefits, including the ability to pave in cooler temperatures, haul longer distances, decrease the compaction effort, and the ability to incorporate RAP material at reduced temperatures

2.2.1 Groups of WMA technologies

There have been a number of products and processes introduced to the market place to produce WMA over the last ten years, which can be categorised in three main groups: organic additives, chemical additives and foaming processes. A brief description of several of these processes is presented here:

- Organic additives that are typically waxes or amides with melting points above 45ºC. At temperatures above the melting point, these materials show a decrease in the binder's viscosity making it possible to produce asphalt mixtures at lower temperatures. In addition, some studies suggest that when added to the binder, these materials improve its lubrication capability resulting in an improvement in mix workability at lower temperatures (Perkins 2009). These mechanisms (i.e. viscosity reduction and lubrication improvement) make it possible to produce asphalt mixtures at lower temperatures. In Europe, the most common organic additives used to produce WMA include Sasobit, Asphaltan – B and Licomont BS-100.

- Chemical additives that usually include a combination of emulsification agents, surfactants, polymers and additives to improve coating, mixture workability, and compactability, as well as adhesion promoters (anti-stripping agents). The most commonly used product in this group of products is Evotherm. This chemical material is stored at 80ºC and makes up 30% mass of the bitumen. It decreases the viscosity of the binder at lower mixing temperatures, which leads to fully coated aggregates at the same temperature (Sargand et al., 2009).

- Foaming technologies that introduce a certain amount of water into the asphalt mixture to generate a foaming effect in the bitumen, expanding its volume and reducing its viscosity providing appropriate workability and coatability conditions at reduced temperatures (D'Angelo et al., 2008). These foaming technologies include: 1) the addition of an additive known as zeolites, 2) the incorporation of wet fine aggregates (Low Energy Asphalt, LEA), or 3) a two component bitumen system (WAM-Foam), which includes mechanical foaming. In terms of the first process, zeolites are mineral additives that contain large vacant spaces in their structures that allow the presence of relatively large molecules and cation groups, such as water (Corrigan, 2008). When added to the asphalt mixture, they release water in the form of steam, thus foaming

the liquid bitumen (Perkins 2009). In the LEA process, an adhesion additive is incorporated into the bitumen, after which it is blended with heated coarse aggregates and wet fine aggregates (i.e. sand) to generate foam (D'Angelo et al. 2008). Finally, in the WAM-Foam process, one of the most common processes used in Europe, a soft bitumen (typically 20-30% of the total bitumen), is first used for coating the aggregates, minus any filler. Then a hard bitumen is added to the mixture using a mechanical foaming technique with special equipment. In this case, coating the coarse aggregates with the soft bitumen contributes to the full coating of these mineral particles, which may not otherwise occur when using a stiffer binder at low temperatures (D'Angelo et al. 2008).

The next section describes a brief history about foaming technologies, as they constitute the main focus of this study, and also they represent one of the most used technologies to produce WMA mixtures.

2.2.2 Brief history and applications of foaming technologies

The process of foaming bitumen was developed more than 50 years ago by Csanyi (1957) of Iowa State University. The availability of ungraded marginal loess in this state and a shortage of good quality aggregate, were the basis for Csanyi's discovery (Csanyi, 1957). This application was first studied for bituminous paving mixtures and for bituminous soil stabilization. For the latter, Csanyi discovered that foamed bitumen could be mixed with ungraded local aggregates (i.e. gravel, sand and loess) to improve their properties and produce a good quality material for paving applications (Csanyi, 1957).

The properties of the produced foamed bitumen through this technique included reduced viscosity at lower temperatures that provided an easy and more uniform distribution of the bitumen during the production of the mixture, and a change in the surface tension of the bitumen to enable coating of the aggregates more readily, in order to improve the adhesion between the aggregate and the bitumen (Csanyi, 1957). In addition, according to Bowering and Martin (1976), these properties of the hot foam enabled coating of moist, cold aggregate surfaces particularly the fine portion of the material.

As means of producing foamed bitumen, various systems were also used in that time such as, water-based, air-based and gases-based. However, the use of steam was proven to be the simplest, most effective and efficient way to produce foamed bitumen (Csanyi, 1957, Sunarjono, 2008). This system, using steam as a foamant, was appropriate for in-plant mixing where steam was readily available, but for in-situ operations due to the need for specialized equipment (i.e. steam boiler) it was proved to be

very impractical (Muthen, 1998). Thereafter, in 1968 Mobil Oil Australia acquired Csanyi's patent so the original foaming process was modified two years later. In the new process, a controlled flow of cold water (rather than steam) was introduced into a hot asphalt stream to produce the foamed bitumen and then it was mixed with cold, wet aggregates or soil (Newcomb et al., 2015). Thus, the bitumen foaming process became more practical for general use (the necessity for a steam generating system was eliminated) and it gained more recognition. In 1976 it was reported that in Australia considerable experience had been gained in the construction of pavements incorporating foamed bitumen to full-scale paving projects (Bowering and Martin, 1976, P. J. Ruckel et al., 1980). This technique was used in various applications such as in the stabilization of sand, gravel and crushed stone aggregates and the production of road courses and sub-base courses for both local roads and heavy traffic roads (Sunarjono, 2008). The use of foamed bitumen stabilization technology then gained acceptance by many road authorities in Europe, South Africa (Acott, 1979., Engelbrecht, 1999) North America (Abel and Hines, 1979), Asia and Australia (P. J. Ruckel et al., 1980).

In 1999 (Jenkins et al.) introduced a new process involving a half-warm foamed bitumen treatment. The authors explored the concepts and possible benefits of heating a wide variety of aggregates to temperatures above ambient but below 100°C before the application of foamed bitumen and they found that preheating aggregates enhanced particle coating, mix cohesion, tensile strength, and compaction. The authors highlighted that this technique was particularly beneficial for mixes containing RAP or densely graded crushed aggregates. In the NCHRP Project 9-53, Newcomb et al. (2015) produced foamed bitumen mixtures for warm and hot applications (i.e. higher temperatures than the previous applications). The authors found that higher production temperatures involved in the foaming process were able to produce foamed asphalt mixtures with better workability and coatability properties.

Furthermore, the foaming processes that form part of the WMA technologies, were developed in Europe in the late 1990s, as a measure to reduce greenhouse emissions. In 1995, the first tests conducted by means of zeolite foaming technologies were reported by MHI (Mitteldeutsche Hartstein-Industrie AG), using Aspha-min® zeolite (Sargand et al., 2009). In the same year, Shell Bitumen filed a patent to cover a WMA technique that used a two-component technique. Koenders et al. (2002), of Shell Global Solutions, described an innovative WMA process that was tested in the laboratory and evaluated in large-scale field trials (in Norway, the United Kingdom, and the Netherlands), highlighting the applications in the production and placement of dense-graded wearing courses (Button et al., 2007). This work of Shell, later in 2002, resulted in the development of WAM-Foam®. In 1999, the first WAM-Foam® trial was placed in Norway and the earliest zeolite trials on public roads were placed in Germany (Prowell et al., 2012). In 2007, Advera® zeolite was introduced by PQ Corporation. Between 2008 and 2010, there was a considerable increase in the number of

WMA foaming technologies in the US, especially the mechanical foaming technique. According to a survey done by the National Asphalt Pavement Association (NAPA) (Hansen and Copeland, 2013), mechanical foaming units were responsible for about 88% of all WMA produced in 2012. Today, both groups of foaming techniques are widely used in paving applications and they represent the largest segment in WMA production (Newcomb et al., 2015).

2.2.3 Bitumen foaming process

In this section, the two foaming processes used for the production of the foamed bitumen mixtures evaluated in this study are described. These technologies are within the most commonly used foaming technologies as was described in the previous section.

The first technology comprises the mechanical foaming technique. The procedure to manufacture foamed bitumen by this technique consists of injecting pressurised air and small quantities of water into a hot bitumen in an expansion chamber. In this process, the injection of water to the hot bitumen causes the liquid bitumen to foam and expand its volume. When the injected water, on contact with the hot bitumen is transformed into vapour, thousands of tiny bitumen bubbles are formed. This process is followed by a rapid collapse of the foam and a slow, asymptotic return of the bitumen to its original volume (Jenkins et al., 2000, Sunarjono, 2008, Wirtgen, 2010).This process is generally performed in the laboratory using a foaming plant. Figure 2.2, illustrates the mechanical foaming process, and Figure 2.3 a foamed bitumen sample produced with this technology.

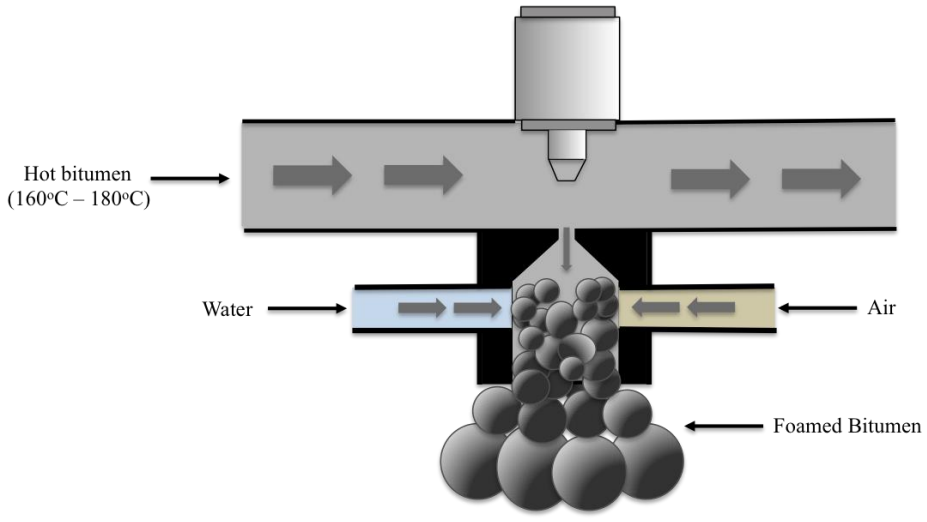


Figure 2.2 Foamed bitumen production by the mechanical foaming technology. After (Wirtgen, 2010)

In its foamed state, the bitumen has lower viscosity and expanded volume up to 20 times its original volume and can therefore be mixed with cold (and often damp) aggregates, which constitutes one of

its main applications. These two conditions allow bitumen in its foamed state to coat the aggregates at lower temperatures in comparison to those required in regular HMA materials. However, for this study foamed bitumen produced with this technology will be combined with aggregates heated at 90, 120 and 160ºC, and also with RAP material, the latter which constitutes another main application of this technology (i.e. for cold processes mainly).

Figure 2.3 Foamed bitumen produced by the mechanical process

The foaming characteristics of the bitumen are usually quantified through two parameters: expansion ratio (ER) and Half-life (HL) (Muthen, 1998, Fu, 2009). ER is the ratio between the maximum volume of foam achieved to the original volume of the binder and, the HL is the time that the foam takes to collapse to half its maximum volume. In general, increasing water content increases the foam volume achieved but it results in a quicker collapse of the foam. Thus, the goal during the foaming process is to achieve a maximum expansion ratio with the maximum possible HL.

These two experimental foam characteristics can be investigated using a laboratory foaming machine. The foamed bitumen produced is collected in a special steel measuring cylinder and then its maximum increased volume is measured by a dipstick (Sunarjono, 2008). The ratio of this value to the original bitumen volume (known) is calculated as ERm, whereas the time needed by the foam to collapse to half its maximum volume is recorded as the HL. Figure 2.4 illustrates the testing method to measure ERm and HL (Sunarjono, 2008).

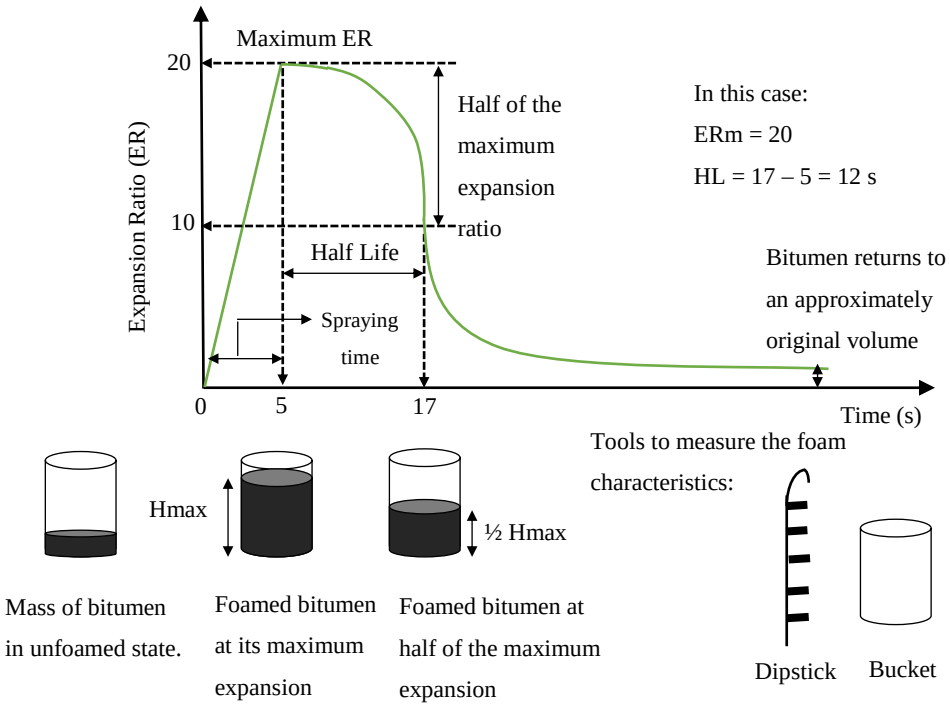


Figure 2.4 Illustration for measurement of maximum expansion ratio and half-life. After (Sunarjono, 2008, Wirtgen, 2010)

The second technology comprises the use of zeolites, which are hydro-thermally crystalized silicates containing large vacant spaces in their structures that allow the presence of relatively large molecules and cation groups, such as water (Hurley and Prowell, 2005). The percentage of water held internally by the zeolite varies between 18 – 21% by mass. These materials are at equilibrium at this moisture content, therefore, it takes significant energy, (i.e. temperatures above 100^{o}C), to release the water. When mixed with the bitumen (i.e. which is usually 20-30^{o}C lower than the hot bitumen used for traditional HMA), they release water gradually in the form of steam, producing micro-bubbles on the bitumen's surface. Figure 2.5 illustrates a foamed bitumen sample produced by this technology. This condition decreases the binder's viscosity and enhances the workability of the asphalt mixture (PQ Corporation, 2012). With this technology, mixture production and placement temperatures are 10-25^{o}C lower than those required for HMA mixture (PQ Corporation, 2012).

The NCHRP Report 691 (Bonaquist, 2011) states that zeolites can be added into the mixture in the mixing bowl, or alternatively, they could be thoroughly pre-blended with the bitumen shortly prior to mixing using a mechanical mixing device.

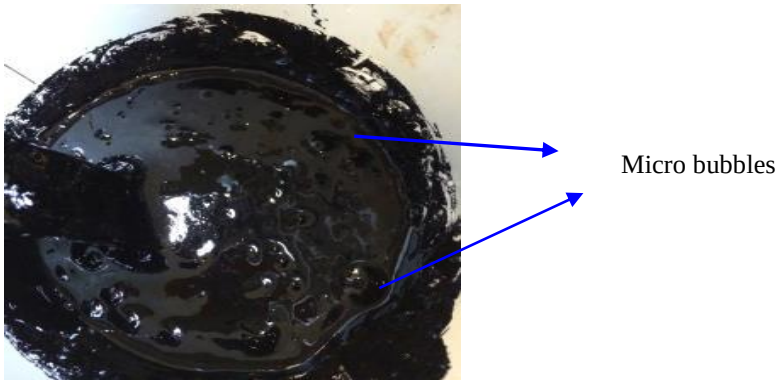


Figure 2.5 Foamed bitumen produced by the addition of zeolites

2.2.4 WMA Moisture Susceptibility

Despite the environmental and possible cost saving benefits of WMA, there are several factors that are related to the lower production temperatures of the WMA and the use of foaming technologies that add water directly (e.g. mechanical foaming) or indirectly (e.g. zeolites) to the asphalt mixture that could increase the moisture susceptibility of WMA. These factors include (Epps et al., 2014, Caro et al., 2012):

- The effects that WMA additives may have on the quality of the adhesion between the bitumen binder and the aggregate,
- The effects of incomplete aggregate drying during the WMA fabrication process or the incorporation of wet, damp aggregates,
- Introduction of additional moisture with the WMA technologies that introduce water to produce foamed bitumen
- Reduced binder absorption by the aggregates at lower production temperatures, and
- Reduced binder-aggregate coating

Bonaquist (2011), evaluated mix design practices for WMA through laboratory and field study, and indicated that the effect of WMA processes on moisture susceptibility is mixture and process specific. The author indicated that different WMA have different effects on moisture susceptibility and that most of them provide the mixture with less resistance to moisture damage, with some exceptions exhibiting better moisture susceptibility. Thus, moisture susceptibility of WMA mixtures should be evaluated comprehensively.

Most of the published works directed to evaluate moisture damage susceptibility in WMA mixtures have used the Hamburg Wheel Tracking test, Tensile Strength Ratio (TSR), Indirect Tensile Strength (IDT) in dry- and wet-condition, and dynamic modulus, among others. With few exceptions,

researchers have reported that moisture causes larger reductions in most mechanical properties of WMA mixtures as compared to those of HMA mixtures. Based on this analysis approach, researchers have suggested that WMA mixtures are more prone to moisture damage than conventional HMA mixtures.

Hurley and Prowell (2005) evaluated the stiffness change by means of resilient modulus, and moisture susceptibility of WMA mixtures which included the incorporation of zeolites. For the former, the authors noted that the addition of zeolite to the bitumen did not significantly affect the resilient modulus of the produced mixtures. The authors concluded that the addition of zeolites does not significantly increase or decrease the strength of hot mix asphalt for any compaction temperature. For the latter, the authors produced Tensile Strength Ratio (TSR) test samples and obtained reduced tensile strength values, and observed visual stripping in the control and the zeolite containing mixes produced at 121°C, indicating that the low compaction temperature used when producing warm asphalt with the incorporation of zeolites, could potentially increase the potential for moisture damage. According to the authors, incomplete drying of the aggregate and resulting water trapped in the coated aggregate were the two possible causes for this effect. Various anti-stripping agents were evaluated to mitigate the potential for moisture damage, and hydrated lime appeared to be effective with granite aggregates.

Similar results were obtained by Xiao et al. (2009a), where a laboratory investigation to evaluate moisture damage on WMA foaming mixtures based on zeolite technology containing moist aggregates was performed. Results from the tests for the Indirect Tensile Stress showed that WMA additives does not negatively affect the dry ITS values for the samples studied. However, an analysis on wet ITS values showed that the addition of zeolites to produce WMA, without anti - strip additives diminish the resistance of the mixture to undergo moisture damage. According to ITS values and TSR results, the addition of anti-strip additives, such as hydrated lime to the mixture has a remarkable effect on moisture damage susceptibility.

Another research on moisture susceptibility on WMA mixtures including foaming technologies was conducted by W. S. Mogawer et al. (2010), based on the concern about the negative effect of warm mix additives on moisture susceptibility, due to the possibility of improper drying of the aggregates and the introduction of water into the mixture. The laboratory investigation included the analysis after the Hamburg Wheel Tracking Device (HWTD), and the stiffness ratio under wet and dry conditions. Results from HWTD suggest that the inclusion of anti- strip agents might reduce WMA susceptibility to undergo moisture damage. Dynamic modulus test results indicated that WMA technologies had no adverse effect on moisture susceptibility. The authors found that from all the evaluated WMA

technologies, the mixtures containing Advera® zeolite presented the highest susceptibility to moisture damage.

Alossta et al. (2011) evaluated the moisture sensitivity of foamed based WMA mixtures from field cored specimens as well as laboratory specimens compacted at two different temperatures (154°C to 132°C). The authors found that the WMA mixtures have slightly comparable moisture resistance compared to the control HMA mixture when compacted at the lower temperature (132°C). However, the authors question the validity of collecting field WMA mixtures, re-heating and compacting them in the laboratory since the effectiveness of the additive could be demolished in the process.

Zeiada et al. (2017) evaluated the laboratory performance of a foamed-based WMA mixture taken from an actual field section in Arizona, USA. The laboratory testing program included the dynamic modulus test of specimes compacted at 154ºC and 132ºC to investigate wether re-heating samples in the lab had an effect on the resistance properties of the material, and also moisture sensitivity by means of AASHTO T-283 tensile strength ratio (TSR) test and the Hamburg wheel-track test. For the former, the results indicated that the reduction in temperature from 154ºC to 132ºC did not reduce the modulus values for the foaming based WMA mixtures. The moisture sensitivity test results based on the TSR values indicated that theses mixtures may have problems with the moisture damage, which was contradicted with the Hamburg wheel-track test results. The authors attribute these differences to the fact that the AASHTO T283 includes both freezing and thawing conditions. Which can be more damaging compared to the Hamburg wheel-track test.

2.3 Reclaimed Asphalt Pavement (RAP)

RAP material is essentially flexible pavement that has reached the end of its service live and is reclaimed to be used as part of new or rehabilitated road structures. In its most common form it is usually collected in loose granular form by milling, planning, or crushing the existing pavement (Figure 2.6). This material can be incorporated as part of hot mix asphalt (HMA) mixtures because its components – the bitumen and aggregates – still have value. The use of RAP in new mixtures can reduce the amount of new material that has to be added, generating possible cost savings and contributing to the conservation of natural resources (Copeland, 2011). Undoubtedly, these factors make this practice a valuable alternative for the construction of road infrastructure projects.

Figure 2.6 RAP material

2.3.1 Importance and challenges of incorporating RAP into hot asphalt mixtures

Despite the great potential RAP material has shown to be reused in asphalt mixtures, typical values of RAP allowed in the fabrication of new asphalt mixtures are limited to a range of 10–20% (Mogawer et al., 2012). Concerns related to the contribution of the hardened bitumen from RAP in the final mixture performance and durability in the field have limited the production of new HMA with higher amounts of this material. One of the reasons for this limitation is related to the unclear role that the bitumen from RAP plays in the presence of virgin materials. During the service life of the road from which the RAP is obtained, the bitumen in the road becomes aged or hardened by reacting with oxygen in the air. This ageing process causes an increase in its viscosity, and subsequent stiffening of the asphalt mixture (Bell, 1989). As a result, there is a common belief that the use of this material can lead to poor field performance especially because the increased brittleness could negatively affect its fatigue and thermal cracking resistance. This belief has been supported by some authors (Bonaquist, 2005, Shah et al., 2007, Daniel et al., 2010), and refuted by others (Hajj et al., 2007, Maupin, 2008, Tabakovi´c et al., 2010). In this regard, to account for the properties of the hardened bitumen in the final mixture, softening agents or rejuvenating agents are commonly added (Sondag et al. 2002). Softening agents lower the viscosity of the aged bitumen, while the rejuvenating agents attempt to restore the properties of the aged bitumen (Roberts et al. 1996). To carry out this task, in Europe, the standard EN 13108-8:2005 for reclaimed asphalt establishes that if RAP content is higher than 10% for surface layers, and higher than 20% for base layers, a logarithmic blending law for penetration and a linear blending law for softening point should be applied to select the proper virgin bitumen to use.

Moreover, another concern related with the use of RAP is associated with the unclear interaction or level of blending between the aged RAP bitumen and the virgin bitumen. In general, there are three possible levels of interaction between the aged and virgin binders (McDaniel & Anderson 2001): 1)

black rock (no blending occurs), 2) total blending (100% blending) and, 3) actual practice. If RAP acts like a black rock, the aged and virgin binders will not interact. Hence, it is assumed that RAP will not significantly change the properties of the virgin bitumen. However, it is usually assumed that RAP does not act as a black rock and that the aged bitumen blends with the virgin bitumen during mixing. In fact, many design procedures, including the European blending laws design method assumes that all the aged bitumen is fully available in the mixture and would effectively contribute to the blend. This means that the amount of virgin bitumen can be reduced by the full amount of bitumen in the RAP for the percentage specified (Al-Qadi et al., 2007). Other researchers state that the amount of blending that actually occurs between the old and the new bitumens is somewhere between complete blending and the black rock behaviour (Copeland 2011).

In this regard, several studies have found that the hardened RAP binder has some effect on the resultant bitumen grade of the final mixture depending on the amount of RAP material that is added, and also that the use of a soft bitumen grade mitigates this stiffening effect. At low RAP content (up to 20%), the change may be negligible. However, further increase in RAP contents may have a clear effect on the bitumen grade. For instance, the NCHRP Report 752 (West et al., 2013) showed that in mixtures containing 55% RAP, the stiffness of the resultant mixture could increase between 25 to 65% compared to virgin HMA (no RAP material). McDaniel et al. (2012) conducted a comprehensive study on plant produced mixes with varying RAP content (0, 15, 25 and 40%) and found that an increase in the RAP content generally caused an increase in dynamic moduli of the mix, although the increase was not statistically significant. The authors also evaluated the effect of the increase in RAP content using a softer grade virgin bitumen, and found that the mix moduli were typically lower than original grade virgin bitumen mixes, and the differences were often statistically significant.

Similar results were presented in the Austroads report (2015). In this report, the authors found that for a given virgin bitumen grade, the increase in percentage of RAP increase the flexural stiffness. It was observed on this study that for mixes with RAP contents of 15% and 30%, the performance properties were very similar, but differed significantly from mixes containing 60% and only virgin bitumen. For mixtures when a softer bitumen grade was used, the addition of RAP produced reasonable results from the stiffness (modulus) perspective for RAP percentages up to 30%.

Furthermore, moisture damage is also one major concern related with the use of RAP hot-mix asphalt mixtures. In general, the presence of water in asphalt mixtures weakens their structural integrity as the bond between the aggregates and the mastic is progressively loss by the presence of this material (Caro et al. 2012). Sondag et al. (2002) conducted moisture sensitivity tests on asphalt mixtures containing different levels of RAP (i.e. 0, 15, 30 and 40%) to determine the moisture susceptibility of these mixtures. The results obtained from this research indicated that mixtures containing those four

levels of RAP did not present considerable moisture damage as quantified by the high values of the Tensile Strength Ratio (TSR) test, conducted following the ASTM D4867 standard. As a conclusion, no correlation was found in this study between the susceptibility to moisture damage and the percentage of RAP used. Loria et al. (2011) also assessed moisture susceptibility by means of TSR test of asphalt mixtures containing 50% RAP content. In this study, the authors included multiple freeze-thaw cycles. The study included field and laboratory-produced mixtures from a provincial highway in Manitoba, Canada. For the former, loose mixtures were sampled during the paving at the project site. For the latter, aggregate, bitumen and RAP source materials were separately collected during production in the plant, and these raw materials were then used to manufacture the laboratory mixes. The TSR results showed that all mixtures exhibited TSR values over 80% after the first cycle, and after the third cycle all mixtures exhibited TSR values lower than the minimum requirement of 80%. Based on these results, the authors concluded that the mixtures containing 50%RAP had an acceptable resistance to moisture damage compared to mixtures with no RAP material or containing low RAP contents of 15%. On the contrary, Li et al. (2004) who evaluated moisture susceptibility on asphalt mixtures containing different levels of RAP (i.e. 0, 20 and 40%), stayed that the susceptibility of the samples to moisture damage was higher for mixtures with higher RAP contents.

At last, RAP variability is also one of the major concerns when working with this recycled material. Such variability includes not only the gradation of the RAP aggregate particles but also the total bitumen content and the bitumen source. Some states have limited the amount of RAP used in paving applications due to this concern. However, some researchers consider that high percentages of RAP could be used if the RAP material is milled off from the same project in which the new asphalt mixture is placed (McDaniel & Anderson 2001). Other researchers have stated that this issue could be controlled by good stockpile control (Roberts et al. 1996). To account for this issue in this study, RAP material will be produced in the laboratory as will be described in the next section.

Although, as described before, several research efforts have been conducted trying to address the major challenges on the incorporation of RAP into HMA mixtures, it is important to highlight that, as demonstrated before, there are still several unsolved questions regarding the actual properties and performance of HMA materials with RAP. Within this context, this research may contribute to get a better understanding on the impact of the incorporation of RAP on the performance of hot asphalt mixtures using a novel and different approach.

2.3.2 Bitumen blending model for mixtures containing RAP

Since RAP material is obtained from pavements that have been in service, the properties of this hard RAP bitumen must be taken into account in the final mixtures. Before this material is re-used, it is required to meet several specifications and regulations which usually vary depending on the country in which this material is going to be used. Thus in order to meet these specifications and particularly when the percentage of RAP in the mix is high, blending models have to be used to design the final content of RAP or to design a final binder with certain desired properties This section describes the bitumen blending model commonly used in Europe, and that was used in this study.

According to the European Standard for reclaimed asphalt EN 13108-8:2005, when using more than 10% of RAP for surface courses and more than 20% for binder courses in combination with virgin bitumen (i.e. unmodified), the penetration in the resulting bitumen blend could be determined through the following equations:

$$Alogpen_1 + Blogpen_2 = (A + B)log_{pen_{blend}} \quad 1$$

where pen_1 is the penetration of the recovered bitumen from RAP; pen_2 is the penetration of the virgin bitumen, pen_{blend} is the penetration of the final bitumen blend in the mixture containing RAP, and A and B are the ratios by mass of the bitumen from the RAP and of the virgin bitumen respectively ($A + B = 1$). Similarly, the softening point of the virgin and RAP blended bitumen can be computed as:

$$T_{R\&B_{blend}} = AT_{R\&B_1} + BT_{R\&B_2} \quad 2$$

where $T_{R\&Bblend}$ is the softening point in the final bitumen blend, $T_{R\&B1}$ is the softening point of the recovered bitumen from RAP and $T_{R\&B2}$ is the softening point of the virgin bitumen, A and B represent the same ratios as in equation (1).

2.4 FAM materials

2.4.1 Different scales for studying bituminous materials

After a thorough review of the relevant literature regarding the materials used in this study, this section describes FAM materials. Initially the different scales of bituminous materials are introduced,

to have an overview of the complete picture. The different scales of bituminous materials, include (from small scale to large scale):

- Bitumen: which constitutes the main element, is a hydro-carbon product produced by removing the lighter fractions (such as liquid petroleum gas, petrol and diesel) from crude oil during the refining process (Hunter et al., 2015).
- Mastic, which is formed by bitumen and mineral filler (particles less than 63 μm)
- FAM materials, which are composed by the combination of bitumen with the finest portion of the aggregates (e.g. particles size smaller than 1 mm) and air voids,
- Asphalt mixture, which is composed by a combination of coarse and fine particles, bitumen and air voids.

Figure 2.7 illustrates the different scales of bituminous materials.

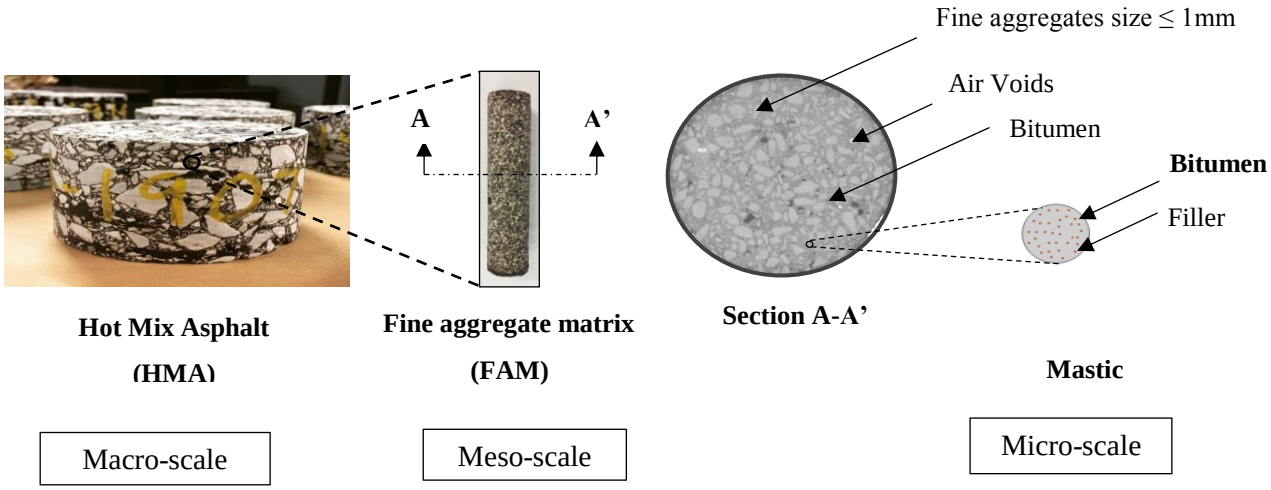


Figure 2.7 Different scales of bituminous materials

FAM materials are one length scale below full asphalt mixtures, at the meso-scale level. While the coarse aggregates in a full asphalt mixture are responsible for the interlock and resistance to the compressive loads that are applied to the asphalt layer, the FAM is what holds the structure together when tensile stresses act on the bottom of the hot mix asphalt layer, providing the appropriate cohesion the otherwise granular material would not have (Cavalcanti De Sousa 2010). The study of this material is particular relevant since it is considered that most damage processes in HMA, especially those related to fatigue cracking and moisture damage, initiate and propagate through this fine matrix (Montepara et al., 2011).

2.4.2 Existing work on FAM materials

Many researchers have used FAM materials to characterise the viscoelastic properties (i.e. linear and non-linear), and gain insight into different deterioration processes (e.g. fatigue, moisture damage) occurring at the macro-scale in asphalt mixtures under various test mode conditions (i.e. torsional and tension compression). As it represents the fine portion present in the full asphalt mixtures, it is expected to represent the behaviour of the full asphalt mixture. Up to date, only one study was found to employ FAM materials including RAP, reinforcing that FAM specimens fabricated with RAP material can be successfully evaluated at the meso-scale.

Masad et al. (2006a) evaluated the moisture susceptibility and fatigue performance of FAM samples prepared using bitumen mixed with fine aggregates of 1.18 mm maximum size. In this work, the authors adapted an analytical non-linear viscoelastic model for characterising fatigue damage in FAM materials, which was initially developed by Lytton et al. (1993) for full asphalt mixtures. The model is based on the cumulative fatigue related damage that the asphalt mixture experiences throughout its service life. The output parameter of the model is the crack growth index, ΔR, which quantifies the average size of the crack length that is generated within the FAM specimens as a function of the number of load cycles (N). The FAM materials were tested using the Dynamic Mechanical Analyser (DMA), which has been shown to be a powerful tool for material characterisation under different loading conditions (frequency, temperature, modes of loading, etc.), and material condition (dry and wet) (Goodrich, 1988, Smith and Hesp, 2000, Kim and Little, 2004). The methodology developed here was able to capture the influence of moisture on the crack growth, and it was also capable of separating the good performing mixes from the poor performing mixes.

In a different study, Masad et al. (2008) evaluated the mode of loading effect on the fatigue damage resistance for different FAM materials which represented conventional HMA mixtures. The authors conducted DMA testing under low strain/stress amplitudes to determine linear viscoelastic properties and high stress/strain amplitudes to determine non-linear viscoelastic properties and dissipated energy associated with damage. The authors derived two fatigue damage parameters based on energy concepts that generated comparable results for both strain and stress controlled conditions. The authors highlighted that the study was conducted on FAM materials, but it could be extended for full-scale asphalt mixtures.

Caro et al. (2008a), used FAM materials representing conventional HMA to characterise moisture damage by applying probability theory to the analysis of the fracture mechanical model, previously used by Masad et al. (2008) within a deterministic framework in FAM materials. The authors also compared the results obtained from the evaluation of moisture damage in FAM materials with those

obtained from the testing of full HMA by the saturation aging tensile stiffness (SATS) test. The results obtained for the fine-graded asphalt mixtures were in good agreement with those obtained for the full scale mixtures by the SATS test, highlighting the use of FAM materials to represent the behaviour in the whole mixtures.

In a different study, Caro et al. (2012), used FAM materials to represent various WMA mixtures and evaluate moisture susceptibility using the crack-growth index model developed by Masad et al. (2008). The authors were able to quantify the effects of moisture on the mechanical response of the WMA fine mixtures and rank them according to their performance.

Tong et al. (2013), used FAM materials to evaluate the fatigue crack growth affected by water vapour diffusion and ageing in conventional HMA mixtures. The authors developed a method of predicting and quantifying the amount of water that can enter into the surface asphalt layer by vapour transport, and used a different approach of the DMA testing technique, by developing a repeated direct tensional test method for cylindrical FAM specimens. For the fatigue characterisation, the authors analysed the crack propagation which occurs in the 'tertiary stage' of the direct tension test proposed. According to the authors, in this stage the modulus magnitude decreases and the phase angle increases with increasing load cycles, indicating the opening and propagating of cracks in the specimens. The authors were able to evaluate the extent to which fatigue crack growth in a pavement structure would result from such moisture accumulation, and highlighted the efficiency of the method for fatigue crack growth characterisation in these materials.

Gudipudi and Underwood (2015), studied the relationship between FAM materials and full asphalt mixtures by applying tension-compression loading tests for both dynamic modulus and fatigue damage characteristics. In terms of the dynamic modulus, the authors found from the corresponding master curves for both materials, a similar frequency dependence in both material cases. Furthermore, in terms of fatigue analysis, the authors found that FAM materials resisted higher strains than the full asphalt mixtures for the same fatigue life. The authors concluded that testing FAM materials in tension-compression loading tests, leads to similar comparisons between the two materials, and highlight the potential use of FAM mixtures for material characterisation and ranking mixtures.

He et al. (2016) reported for the first time, the use of FAM materials as an alternative approach to characterise blended binders in asphalt mixes containing RAP and RAS (Recycled Asphalt Shingle) materials. The authors determined the linear viscoelastic properties of FAM materials prepared using virgin aggregate, virgin bitumen, RAP, RAS, and a rejuvenating agent, by means of DMA testing. Based on the results of this study, the authors concluded that FAM testing can be considered to be an effective alternative approach to chemical binder extraction for characterising the properties of

blended binders, compatibility of virgin binders with RAP and RAS binders, and the influence of rejuvenating agents.

2.5 Viscoelastic nature of bitumen and FAM materials

Asphalt mixtures are viscoelastic materials, which means that their response under load depends upon the rate of loading and the temperature. The viscoelastic nature of an asphalt mixture comes primarily from the viscoelastic behaviour of the bitumen. Depending on the temperature and rate of loading, bitumen could behave as an elastic material or as a viscous material, or in a way that reflects both its elastic and viscous nature. At low temperatures and/or during rapid loading (short loading times – high loading frequencies) it behaves like a glass-like elastic solid, whereas at high temperatures and/or during slow loading (long loading times – low loading frequencies) the response is as a viscous fluid (Airey, 1997).

The dependence of asphalt mixtures and bitumen properties with time and temperature can be studied through rheology. Rheology is the study of materials' flow and permanent deformation under applied forces. The word rheology is believed to come from the Greek words "ρεω", which means "to flow", and "λογοο" meaning "word, science" and, therefore, it literally means "the study of the flow" or "flow science" (Barnes H A, 1989, Airey, 1997).Understanding the flow and deformation (rheological properties) of bitumen in an asphalt mixture is crucial for pavement performance. Asphalt mixtures that deform and flow too readily may be susceptible to rutting, while those that are too stiff may be prone to fatigue and cracking (Yussof, 2012).

The rheological properties of bitumen can be measured by means of an oscillatory type testing known as dynamic shear rheometer (DSR), generally conducted within the linear viscoelastic response. For the case of FAM materials, their rheological properties can be measured by a similar dynamic test, usually known as dynamic mechanical analysis (DMA), which consists of using a rheometer with a special solid fixture configuration to conduct shear controlled strain tests on the small cylindrical FAM specimens. These testing techniques allow the linear viscoelastic properties ($|G^*|$ and δ) of both materials, (i.e. FAM and bitumen) to be determined by a wide range of frequencies and temperatures. The next section describes the general principles of dynamic testing, and then the operational procedure used in the dynamic testing for bitumen and FAM materials are described.

2.5.1 Dynamic response of asphalt materials

In the dynamic test, a sinusoidal strain is applied to a specimen and the resulting stress is monitored as a function of frequency ω (rad/sec). The sinusoidal strain can be represented by (Anderson et al., 1994, Airey, 1997):

$$\gamma^* = \gamma_0 sin\omega t \tag{3}$$

where, γ^* represents the dynamic oscillating shear strain, γ_0 the peak shear strain, ω the angular frequency (rad/sec), t is the time (seconds). The loading frequency, ω, also known as the angular frequency or rotational frequency, is expressed as:

$$\omega = 2\pi f \tag{4}$$

where *f* is the frequency (Hz) at which the test was measured.

The sinusoidal strain can also be presented in complex notation as:

$$\gamma^* = \gamma_0 e^{i\omega t} \tag{5}$$

where $i = \sqrt{-1}$

The response of the applied strain is a stress which for the linear viscoelastic region (small strains), is sinusoidal and out of phase with the applied strain by an amount δ. The response stress is represented by:

$$\sigma^* = \sigma_0 e^{i(\omega t+\delta)} \tag{6}$$

where σ^* is the dynamic oscillating shear stress (Pa), σ_0 is the peak stress (Pa), and δ is the phase angle (^{o}).

The Phase angle, δ, can be defined as the phase difference between stress and strain. For a perfectly elastic material, there is an instant response and, therefore, the time lag between the applied stress and the resulting strain or phase angle δ is zero. For purely viscous materials instead, the δ is 90^o (Roberts et al., 1996). Thus, the phase angle is important in describing the viscoelastic properties of a material such as asphalt cement (Anderson et al., 1994).

The sinusoidal, oscillatory, stress and strain waveforms and the resulting dynamic test outputs are shown in Figure 2.8 and Figure 2.9. The ratio of the resulting stress to the applied strain is the complex shear modulus, $|G^*|$, defined by:

$$G^*(\omega) = \frac{\sigma^*}{\gamma^*} = \frac{\sigma_0}{\gamma_0} e^{i\delta} \quad (7)$$

where $G^*(\omega)$ is the shear complex modulus (Pa), as a function of rotational frequency, ω.

The absolute value, or the norm of the complex shear modulus, is the ratio of the peak stress to the peak strain:

$$|G^*(\omega)| = \frac{\sigma_0}{\gamma_0} \quad (8)$$

Equation 8 can also be written as

$$G^*(\omega) = \left(\frac{\sigma_0}{\gamma_0}\right) cos\delta + i\left(\frac{\sigma_0}{\gamma_0}\right) sin\delta \quad (9)$$

Which corresponds to the definition for the complex shear modulus, $|G^*|$, given in the Eurobitume glossary of rheological terms (Eurobitume, 1996).

$$G^* = G' + iG'' \quad (10)$$

Where G* is the complex shear modulus, G' is the storage modulus (Pa), and G'' is the loss modulus (Pa).

The norm of the complex modulus, $|G^*|$ can be calculated from the square root of the sum of the squares of the components:

$$|G^*| = \sqrt{[(G')^2 + (G'')^2]} \quad (11)$$

The in-phase component of G*, or the real part of the complex modulus, is the shear storage modulus, and equals the stress that is in phase with the strain divided by the strain, or:

$$G' = G^* cos\delta \quad (12)$$

The storage modulus, describes the amount of energy that is stored and released elastically in each oscillation and is therefore also known as the elastic modulus, or the elastic component (recoverable part) of the complex modulus (Airey, 1997).

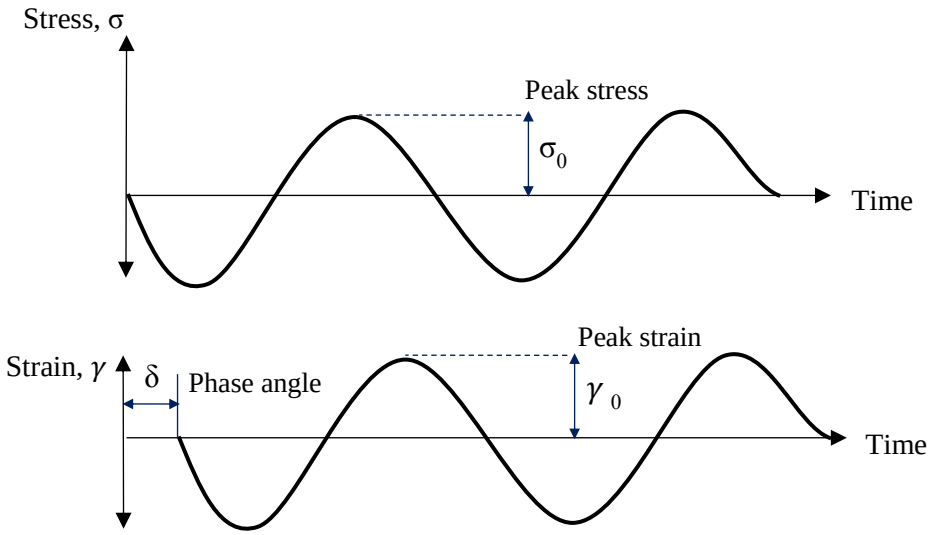


Figure 2.8 Dynamic oscillatory stress-strain functions (Airey, 1997)

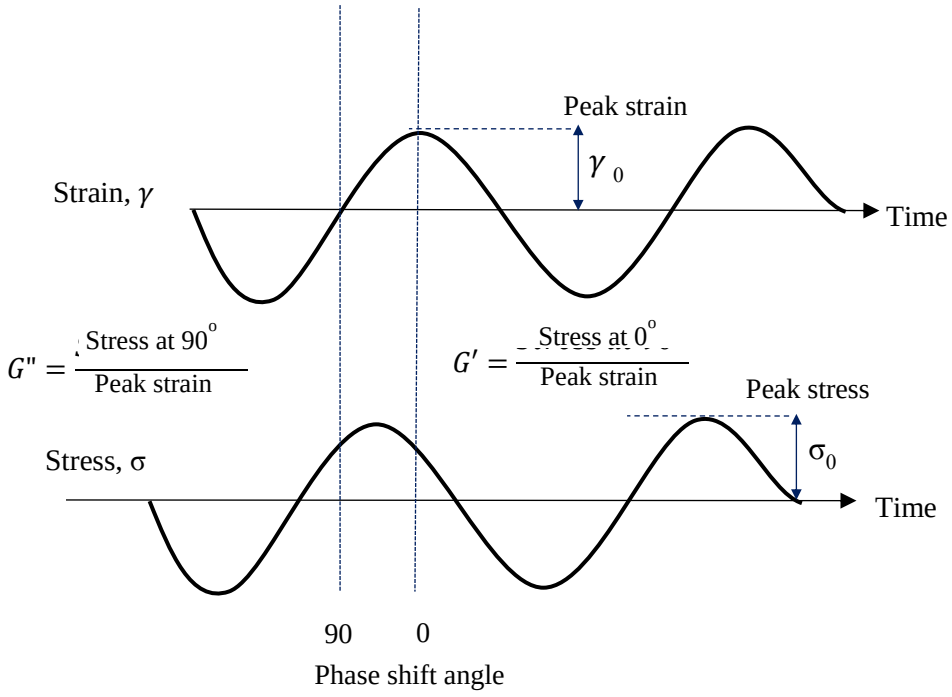


Figure 2.9 Dynamic test outputs from dynamic mechanical analysis (DMA); after Airey (1997)

The shear loss modulus, or the imaginary part of the complex modulus, is the out-of-phase component of G^*, and equals the stress 90° out of phase with the strain divided by the strain, or:

$$G'' = G^* sin\delta \tag{13}$$

The loss modulus, or the viscous (non-recoverable) part, describes the average energy dissipation rate in the continuous steady oscillation found in dynamic test. The loss tangent is defined as the ratio of the viscous and elastic components of the complex modulus or simply the tangent of the phase angle.

$$\delta = tan^{-1}\left(\frac{G''}{G'}\right) \tag{14}$$

The relationships between $|G^*|$, G', G'' and δ are visualized through the vector representation, as shown schematically in Figure 2.10.

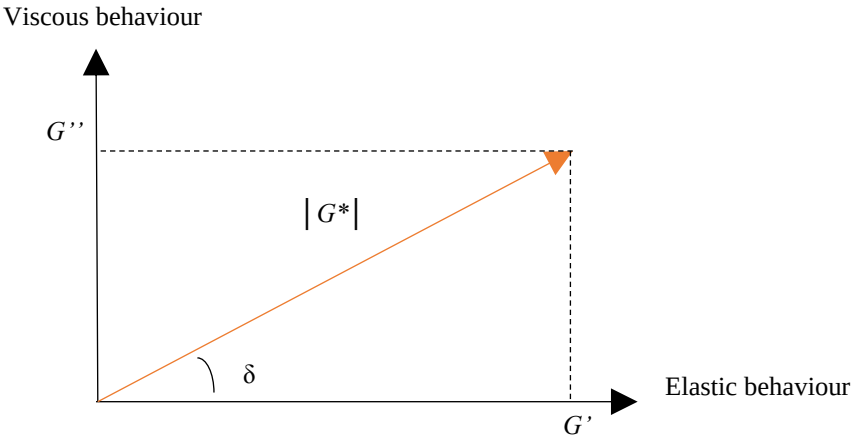


Figure 2.10 Relationship between $|G^*|$, G', G'' and δ after Airey (1997).

The storage and loss moduli are sometimes misinterpreted as the elastic and viscous modulus respectively. In reality, the elastic component of the response only represents part of the storage modulus and the viscous response only part of the loss modulus. In addition, viscoelastic materials exhibit a significant amount of delayed elastic response that is time dependent but completely recoverable. The storage and loss modulus both reflect a portion of the delayed elastic response (Airey, 1997).

The loss of tangent is defined as the ratio of the viscous and elastic components of the complex modulus, or the tangent of the phase angle:

$$tan\delta = \frac{G''}{G'} \tag{15}$$

2.5.2 Dynamic viscoelastic characterisation of bitumen and FAM materials

The operational procedure used with the DSR to measure the linear viscoelastic properties of bitumen samples is to apply sinusoidal, oscillatory stresses and strains to a thin disc of bitumen, which is sandwiched between the two parallel plates of the DSR as depicted in Figure 2.11 Normally the tests are conducted over a range of temperatures and loading frequencies in order to provide a complete characterisation of the viscoelastic properties of the bitumen (Airey and Hunter, 2003).

The amplitude of the responding stress is measured by determining the torque transmitted through the sample in response to the applied strain. Therefore, the stress and strain parameters can be calculated as:

$$\sigma = \frac{2T}{\pi r^3} \tag{16}$$

$$\gamma = \frac{\theta r}{h} \tag{17}$$

where σ is a shear stress, T is a torque, r is radius of parallel discs, γ is shear strain, θ is deflection angle and h is a gap between parallel discs.

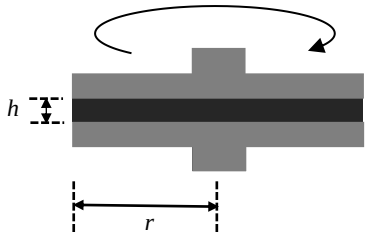


Figure 2.11 Dynamic Shear Rheometer testing geometry

The shear stress and strain in Equations 16 and 17 are dependent on the radius of the parallel discs and vary in magnitude from the centre to the perimeter of the disk. The shear stress, shear strain and complex modulus, which is a function of the radius to the fourth power, are calculated for the maximum value of radius. The phase angle, δ, is measured by the instrument by accurately determining the sine wave forms of the strain and torque (Airey, 1997).

The strains that are applied during the dynamic testing must be kept small to ensure that the test remains in the LVE region. Strain sweeps can be used to verify that testing occurs in the LVE region. In general, the strain must be less than 0.5% at low temperatures but can be increased at high temperatures. Various parallel disc sizes can be used during dynamic mechanical testing. The size of the disc that should be used to test the bitumen decreases as the expected stiffness of the bitumen

increases. In other words, the lower the testing temperature, the smaller the diameter of the disc that needs to be used to accurately determine the dynamic properties of bituminous binders (Goodrich, 1988).

For FAM materials, the general procedure consists of the application of a sinusoidal shear stress or strain to the small cylindrical specimens to measure the corresponding shear strain or stress response respectively (Figure 2.12). This method is commonly known as Dynamic Mechanical Analysis (DMA), and is used to characterize and analyse the response of viscoelastic materials subjected to dynamic loading (Masad et al., 2006b, Castelo Branco, 2008, Caro et al., 2008a, Masad et al., 2008).

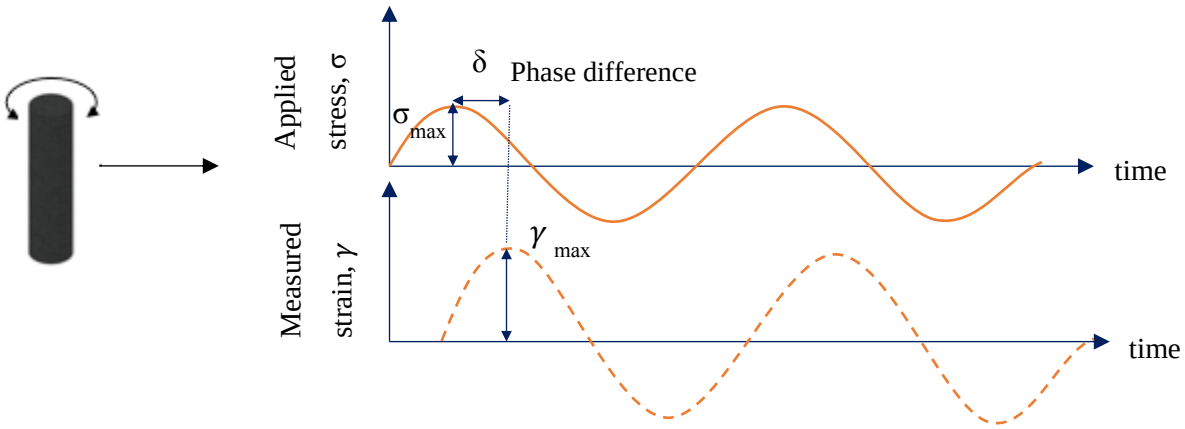


Figure 2.12Dynamic testing in FAM materials

The rheometer used for DMA testing is able to perform different types of testing depending on the scope of the study. In this study, frequency and temperature sweep tests to determine the linear viscoelastic material properties of each type of mixture will be considered

From the measured rheological data at different temperatures and frequencies, a master curve can be constructed as described in the next section. The same principles for the construction of master curves apply for both bitumen and FAM materials.

2.5.3 Construction of dynamic shear modulus master curves

Several researchers have found that there is an inter-relationship between temperature and frequency, which by the use of shift factors, the measurements done at different temperatures can be shifted to fit one overall continuous curve at a reduced frequency or time scale (Monismith, 1966, Dickinson E. J. , 1974, Goodrich, 1988, Airey, 1997). This curve termed as master curve, can be constructed from the results obtained from the dynamic test, and it synthetises the viscoelastic properties (i.e. $|G^*|$ and δ) of the material, at a reference temperature for different values of frequency and temperature.

The principle that is used to relate the equivalency between frequency (time) and temperature and thereby produce the master curve is known as the time-temperature superposition principle (TTSP) (Airey, 1997).

According to Airey (1997), modulus curves at low temperatures crowd together at high frequency/low temperature values, and at very high frequencies they nearly all coincide with one horizontal asymptote (Figure 2.13). At this region, the modulus is called the glassy modulus, Gg. However, under viscous conditions, there is no convergence to a single viscous asymptote as viscosity depends on temperature and therefore each temperature gives rise to a separate viscous flow asymptote.

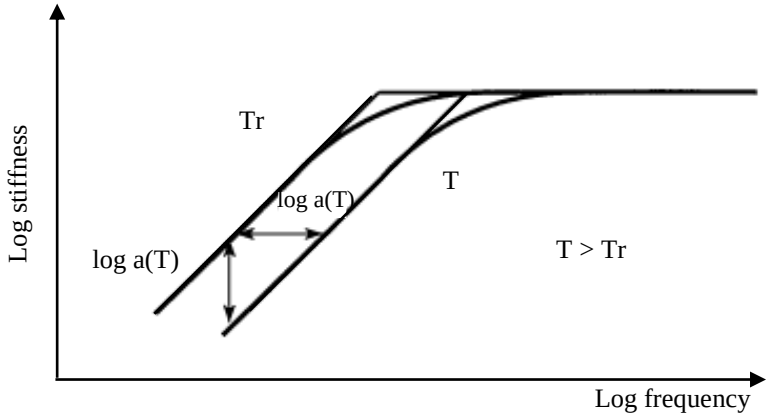


Figure 2.13 Time-temperature superposition principle after (Airey, 1997)

Airey (1997) also noted that because the limiting viscous behaviour is strongly temperature dependent and the elastic behaviour is not, it is possible to separate the influence of frequency and temperature. The concept of time temperature superposition (Figure 2.13), which shows an asymptote pair for an arbitrary reference temperature, Tref (or Tr). If the temperature is increased from Tref to T there is a decrease in viscosity by a factor aT. Therefore, the viscous asymptote at T lies an amount of log aT below that of Tref. However, the elastic asymptote is negligibly changed during the temperature rise. The result is that the asymptote pair appears to be shifted a distance log aT along the log ω axis, because the viscous asymptote has unit slope. The viscoelastic response of a bitumen is a transition between the asymptotic viscous and elastic response and is represented by the curve for Tref. If a change in temperature causes the modulus curve to shift together with its asymptotes over the same distance log aT, the material behaves as a thermo-rheologically simple one (Airey, 1997).

A reference temperature can be chosen and the next higher modulus curve shifted coincides with the reference temperature curve to obtain a value for the horizontal shift factor log aT and a more

extended modulus curve. This procedure is repeated for all curves in succession to obtain a master curve. The effect of temperature on complex modulus is, therefore, to shift the curve of log |G*| versus log ω axis without changing its shape. This permits the reduction of isotherms of log |G*| versus log ω measured over a wide range of temperatures to a single master curve (Airey, 1997).

The extended frequency scale used in a master curve is referred to as the reduced frequency scale and defined as:

$$log_{f_r} = logf + loga_T \tag{18}$$

Where f_r is the reduced frequency (Hz), f is the frequency (Hz) and a_T is the shift factor.

The amount of shifting required at each temperature to form the master curve is called the shift factor, a_T. A log(a_T) plot versus temperature with respect to the reference temperature curve is generally prepared in conjunction with a master curve. This plot gives a visual indication of how the properties of viscoelastic material change with temperature (Anderson et al., 1994).

Because bituminous binders are linear viscoelastic in a wide range of their applications and they are found to be "thermorheologically simple", the time temperature superposition principle can be used to determine their master curves (Airey, 1997).

2.5.4 Rheological data representation

In order to study the rheological properties of bitumen and FAM materials, the rheological data obtained from the dynamic tests can be represented by different plots which help to get a complete picture of the rheological behaviour of the material in different representations. This section describes the most common rheological data representation diagrams, providing an example of the typical diagrams for FAM materials.

Isochronal plots: An equation or a curve on a graph representing the behaviour of the system at a constant frequency (time of loading). For instance, in a dynamic test, curves of complex modulus or δ as a function of temperature at a constant frequency are isochrones (Eurobitume, 1995) .

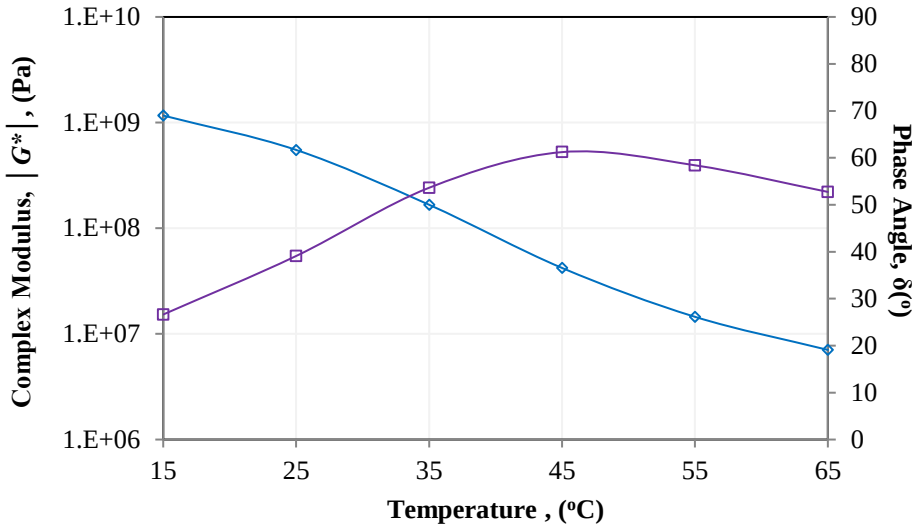
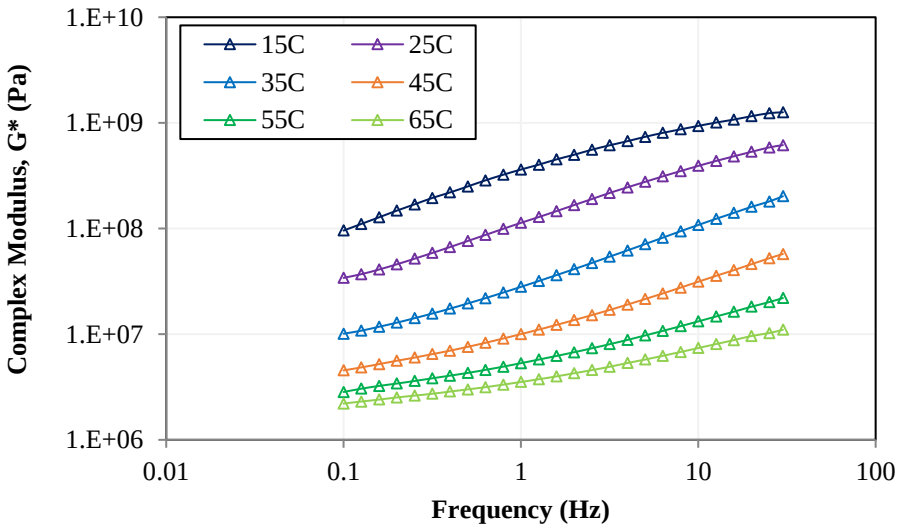
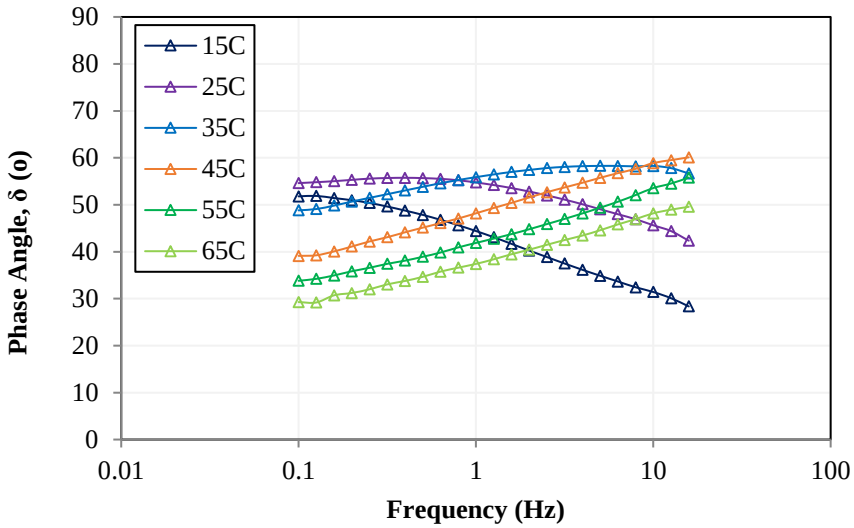


Figure 2.14 Isochronal plots for $|G^*|$ and δ for FAM material

Isothermal plots: An equation of a curve on a graph representing the behaviour of a system at a constant temperature. For example, curves of complex modulus as a function of frequency at constant temperature are isotherms (Eurobitume, 1995).



(a)



(b)

Figure 2.15 Isothermal plots for a) G* and, b) δ for FAM material

Black Diagrams: graph of complex moduli versus phase angles. The frequencies and temperatures are eliminated from the plot, which allows all the dynamic data to be presented in one plot without the need to perform the time-temperature superposition principle (TTSP) manipulations on the raw dynamic shear data. (Airey, 1997).

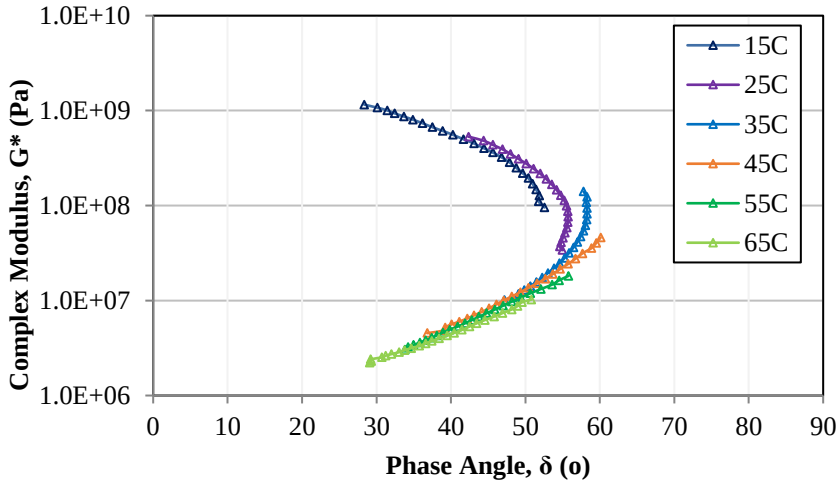


Figure 2.16 Black diagram for FAM material

Cole-Cole Diagrams: A graph of loss moduli as a function of storage moduli. This plot provides a means of presenting the viscoelastic balance of the bitumen without incorporating frequencies and/or temperatures as one of the axes (Airey, 1997).

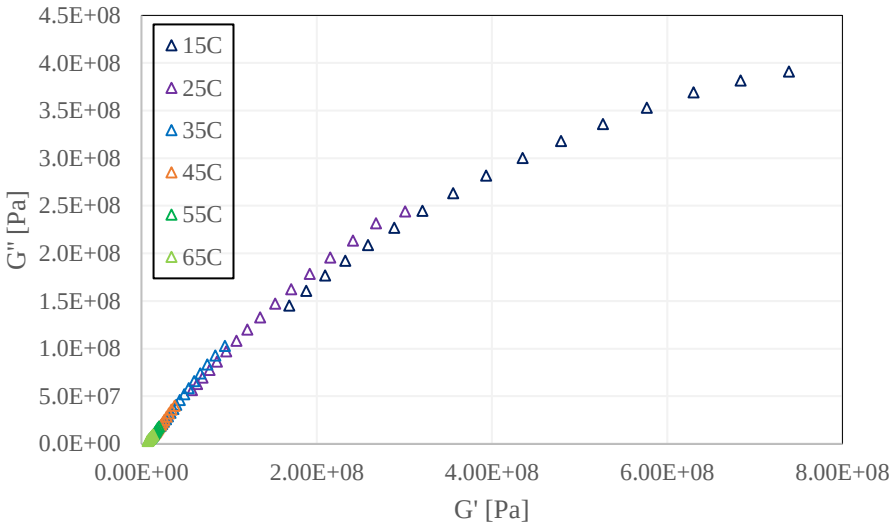


Figure 2.17 Cole-Cole diagram for FAM material

2.5.5 Rheological models for asphalt materials

Another useful tool to describe the rheological properties of bitumen or asphalt mixtures from the experimental data is the use of rheological models. There are several models available in the literature for this purpose (e.g. Dickinson E. J. (1974), Jongepier and Kuilman (1969)) , however in this section only the rheological models used in this study are described.

During the SHRP A-002A research, Christensen and Anderson (1992) developed a mathematical model that described the viscoelastic behaviour of bitumen from the $|G^*|$ master curve. In 1999, Marasteanu and Anderson (1999a) modified this model, referred as Christensen-Anderson-Marasteanu (CAM) Model to improve the fitting particularly in the lower and higher zones of the frequency range of the bitumens. The model is denoted by the following mathematical formulation:

$$G^*(\omega) = G_g \left[1 + \left(\frac{\omega_c}{\omega_r}\right)^{(log2)/R}\right]^{-R/(log2)} \quad (19)$$

where, $G^*(\omega)$ is the complex shear modulus, G_g is the glass modulus (assumed equal to 1 GPa), ω_r is the reduced frequency at the defining temperature (rad/s) , ω_c is the crossover frequency at the defining temperature (rad/s), ω = frequency (rad/s), and R is the rheological index.

Christensen and Anderson (1992) noted that four primary parameters (the glassy modulus, Gg, the steady state viscosity, η0, the crossover frequency, ωc, and the rheological index, *R*) were needed to fully characterise the properties of any bitumen, which are described below (Christensen and Anderson, 1992, Anderson et al., 1994):

1. The Glassy modulus, Gg, is the value that the complex modulus or stiffness modulus approaches at low temperatures and high frequencies (short loading times). Gg is normally very close to 1 ×10 Pa in shear loading for most bitumens and can be used for most purposes.

2. The steady-state viscosity, η0, is the steady-state, or Newtonian viscosity. In dynamic testing, it is approximated as the limit of the complex viscosity, |η*|, as the phase angle approaches 90°. The 45° line that the dynamic master curve approaches at low frequencies is often referred to as the viscous asymptote. It is indicative of the steady-state viscosity, and the value of η0 is binder specific.

3. The cross-over frequency, ωc, is the frequency at a given temperature where tan δ is 1. At this point, the storage and loss moduli are equal. For most bitumens, the crossover frequency is nearly equal to the point at which the viscous asymptote intersects the glassy modulus. The crossover frequency can be thought of as a hardness parameter that indicates the general consistency of a given bitumen at the selected temperature and is binder specific. The crossover frequency is the reciprocal of the crossover time, tc = 1/ωc.

4. The Rheological index, R, is the difference between the glassy modulus, Gg, and the dynamic shear complex modulus at the crossover frequency, |G*(ωc)|. The rheological index is directly proportional to the width of the relaxation spectrum and indicates rheologic type. R is not a measure of temperature but reflects the change in modulus with frequency or loading time and therefore is a measure of the shear rate dependency of bitumen. R is binder specific.

The diagram of a typical master curve is shown in Figure 2.18, illustrating the meaning of these parameters.

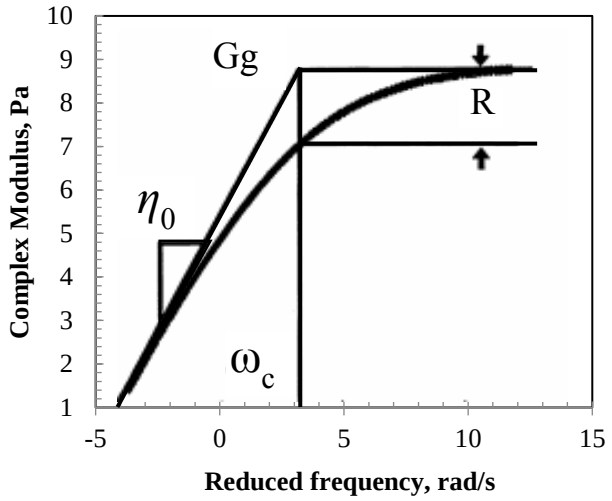


Figure 2.18 Schematic definition of the rheological index (Anderson et al., 1994)

The Gg and ωc can be used in conjunction with the R value to assess the relative ageing and cracking resistance of binders. In this model, the cross over frequency is a measure of the overall hardness of the bitumen (Booshehrian et al., 2013, Christopher Jacques et al., 2015), and its value is lower for aged binders. Furthermore, R value, is an indicator of the master curve shape and to the oxidation of the binder (Booshehrian et al., 2013, Christopher Jacques et al., 2015), and its values are normally high for oxidized binders. Figure 2.19 displays a sample of the $|G^*|$ and δ master curves of a virgin bitumen and the same bitumen after being subjected to an ageing protocol in the laboratory. As the plot shows, ageing results in a flattened $|G^*|$ master curve and a shift of the δ master curve towards lower values, thus, the phase angle value of 45° is obtained for lower values of crossover frequency for the aged bitumen. Besides, the difference between the glassy modulus and the $|G^*|$ at the crossover frequency is higher and thus, the value of *R* increases for the aged bitumen.

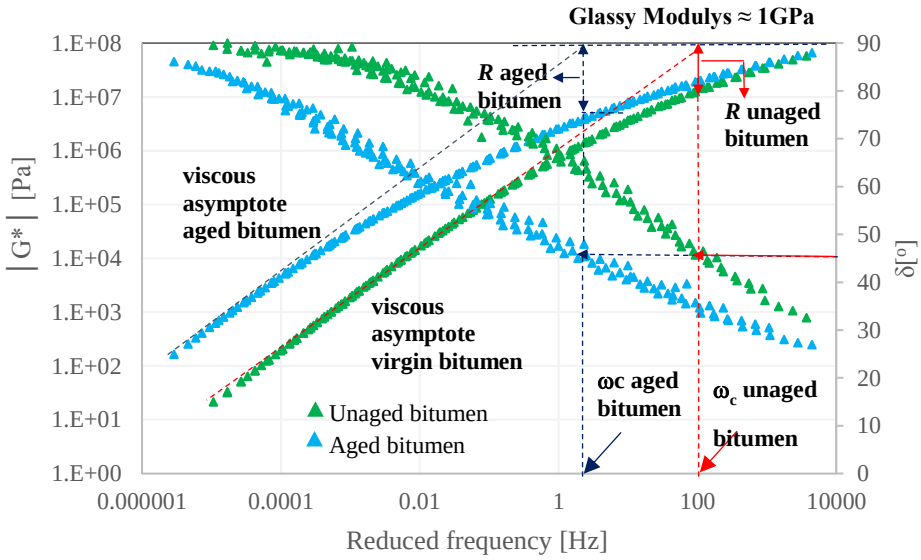


Figure 2.19 A typical master curve for a virgin and aged bitumen and the physical properties

A plot of R-value vs. crossover frequency is usually constructed. Figure 2.20 displays a sample of the R-value plotted against the crossover frequency for the unaged and aged binders shown in Figure 2.19. As the plot shows, the aged bitumen results in a higher R-value, which reflects the flattened master curve, the decrease in cross over frequency, and a wider relaxation spectra as explained previously.

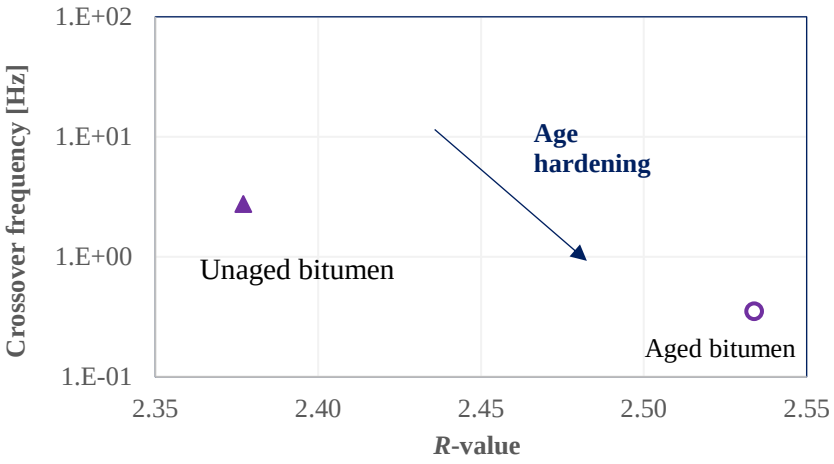


Figure 2.20 Crossover frequency vs. R-value plot

Moreover, in the National Cooperative Highway Research Program (NCHRP) Project A-37A, a new dynamic modulus function, referred as the Sigmoidal Model (Equation 20) was developed to describe the rate dependency of the modulus master curve (Pellinen and Witczak, 2002, Pellinen et al., 2002, Bonaquist and Christensen, 2005, Medani et al., 2004).

$$log|G^*| = \delta + \frac{\alpha}{1+e^{\beta+\gamma\log(\omega)}} \tag{20}$$

where $|G^*|$ is the dynamic shear modulus, ω_r is reduced frequency, and α, β, δ and γ are the fitting coefficients. δ is the lower asymptote, α is the difference between the upper and lower asymptote values, and β and γ define the shape of master curve; γ is the width of relaxation, and β the location of inflection point (the frequency of inflection point = $10^{\beta/\gamma}$) (Rowe et al., 2009).

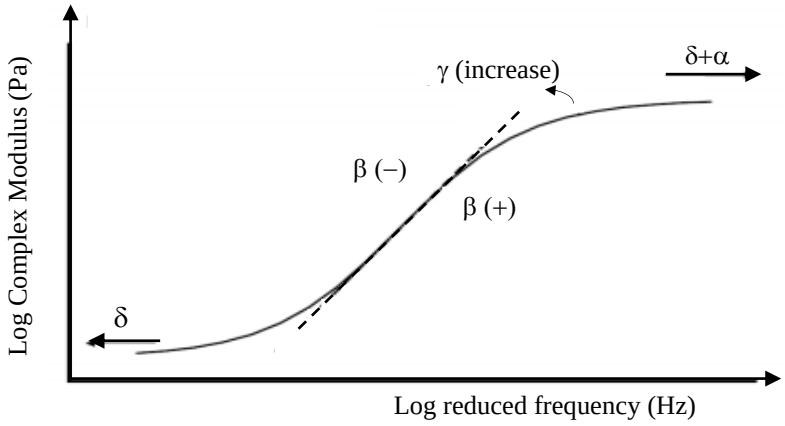


Figure 2.21 Definition of the Sigmoidal Model (Pellinen et al., 2002)

The sigmoidal model has been widely used by many researchers to describe the complex modulus of asphalt mixtures (Pellinen and Witczak, 2002, Pellinen et al., 2002, Bonaquist and Christensen, 2005, Medani et al., 2004, Reyhaneh Rahbar-Rastegar et al., 2017, Mensching et al., 2017). In this study, the Sigmoidal Model is used for describing complex modulus master curve of the FAM materials

The fitting parameters β and γ can be used to assess the relative ageing of asphalt mixtures. Mensching et al. (2016) discussed the impact of these parameters on the changes in the complex modulus master curves as asphalt materials age. To demonstrate this effect, Figure 2.22 shows how the shape of the master curve changes with varying γ and β. The authors described that as asphalt materials age, the master curve flattens through manifestation of a more oxidised bitumen, and γ value increases (the absolute value decreases). The inflection point, which is described by the β parameter, moves to the left (lower frequencies) with ageing or with increased RAP content in asphalt mixtures (Mensching et al. 2015).

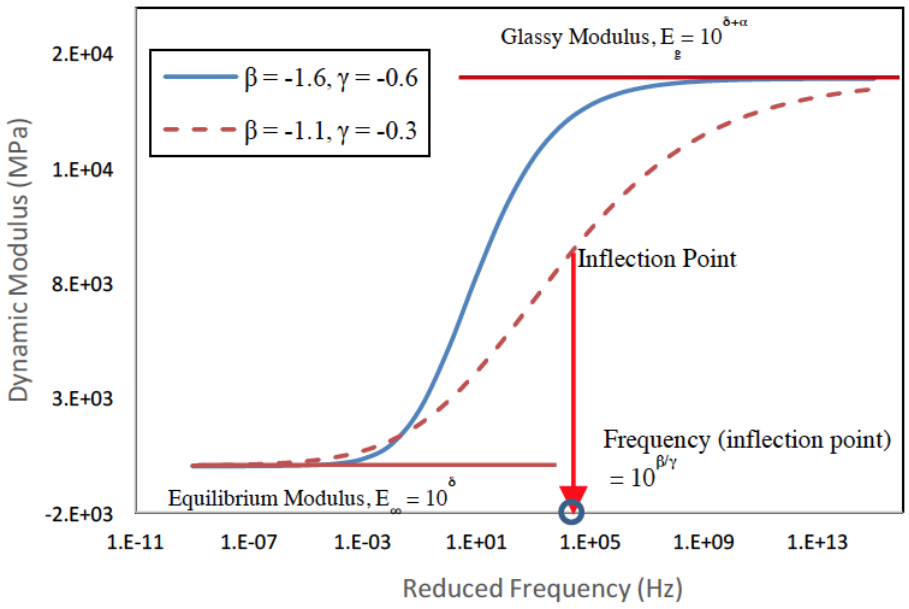


Figure 2.22 Schematic of Dynamic Modulus master curve with varying γ and β parameters (Reyhaneh Rahbar-Rastegar et al., 2017)

2.6 Effects of environmental factors on the performance of asphalt mixtures

During the service life of a road, asphalt pavements have to carry the traffic loads under several weather conditions. In order to satisfy the performance demand, the pavement must have the ability to withstand any damage during the whole service life. According to Scholz (1995) "Durability as it

applies to bituminous paving mixtures is defined as the ability of the materials comprising the mixture to resist the effects of water, aging and temperature variations, in the context of a given amount of traffic loading, without significant deterioration for an extended period".

In the following sections, the two most important damage factors on durability of asphalt mixtures, named moisture damage, and age hardening are described.

2.6.1 Moisture damage

According to Kiggundu and Roberts (1988) moisture damage is 'the progressive functional deterioration of a pavement mixture by loss of the adhesive bond between the asphalt cement and the aggregate surface and/or loss of the cohesive resistance within the asphalt cement principally from the action of water'.

The detrimental effects of water in asphalt mixtures include the adhesive and cohesive types of failure; the former is related to the loss of bond between the aggregate and bitumen interphase, while the latter refers to the loss of resistance within the bitumen-filler mastic of the mixture (Airey et al., 2005, Caro et al., 2008b). The loss of bond caused by the existence of moisture at the binder-aggregate interface or inside the binder, results in the degradation of the mechanical properties of the asphalt mixture, i.e. loss of stiffness and mechanical strength, which ultimately leads to the failure of the road structure (Grenfell et al., 2015).

The occurrence and evolution of moisture damage is considered a complex phenomenon involving chemical, physical, mechanical and thermodynamic processes (Caro et al., 2008b). There are three mechanisms in which the water enters the asphalt mixture and reaches the aggregate – bitumen interface (Masad 2008):

- Infiltration: it is the movement of surface water through the material, which is directly related to the air voids distribution and size due to the porous nature of the medium.
- Capillary rise: it is the rise in a liquid above the level of zero pressure due to a total upward force produced by the attraction of the liquid molecules to a solid surface.
- Diffusion: it is the process by which water proceeding from an underground source or environmental moisture permeates the material.

Several characteristics of the asphalt mixtures affect their moisture susceptibility. Some of these factors include the asphalt film thickness, aggregate characteristics, the air void distribution and

permeability (Masad et al., 2006a). For the first factor, the type of failure is usually associated with the bitumen film thickness covering the aggregates where thin bitumen films generally lead to an adhesive failure, while thick bitumen films are associated with cohesive failure (Lytton, 2005). Furthermore, for the second factor an increased texture and angularity of the aggregates, lowers the potential to moisture damage due to an increase in the total bond energy in the asphalt mixture (Masad et al., 2006a). In terms of the last factor, in general small air void sizes are associated with low permeability and reduction of water infiltration within the mixture, while larger air voids make it easier for the water to drain out of the mixture. In this sense Masad et al., (2006a) stated that there is a 'pessimum range' of air voids between both limits, which maximize moisture damage.

Furthermore, there are several distresses that occur in the asphalt mixtures due to the detrimental effects of moisture, such as (McGennis et al., 1984, Stuart, 1990., Hicks et al., 2003, Caro et al., 2008b):

- Stripping: the process that results in physical separation of the asphalt binder and aggregate due to the loss of adhesion at the interface of these materials in the presence of water or water vapour. Some authors consider stripping a synonym of displacement (Stuart 1990).
- Ravelling: a distress manifestation that is identified by the dislodgement of aggregate particles in the mixture from the surface.
- Shelling: a distress manifestation identified by the loosening and removal of aggregate from a seal coat or other surface treatment.
- Hydraulic scour: a process that occurs on a saturated surface by which the pavement material is eroded due to the dynamic

In addition to moisture-induced damage in asphalt pavements, typical pavement distresses such as fatigue and permanent deformation are intensified by the presence of moisture (Epps et al., 2003).

2.6.2 Ageing

The exposure of asphalt mixtures to various influencing factors during production and service life results in short-term and long-term ageing. The short-term ageing occurs initially during the mixing process of the asphalt mixtures, where their constituent materials are set to high temperatures. In addition to the mixing phase, short-term ageing also occurs during storage, transportation and placing of the asphalt mixture in field (Bell, 1989). The long-term ageing of asphalt mixtures occurs due to their exposure to external weather conditions during their service lives (Bell, 1989). All these factors affect mainly the bitumen present in the asphalt mixtures, resulting in a significant increase in its

viscosity and changes in the associated rheological and physicochemical properties such as the complex shear modulus and adhesion (Roberts et al. 1996).

Age hardening during construction and service has been associated with six major mechanisms (Roberts et al. 1996; Tyrion 2000; Karlsson and Isacsson 2006, AL-Qadi, 2007):

- Oxidation through diffusive reaction between the binder and oxygen in the air;
- Volatilization through evaporation of the lighter components especially during construction;
- Polymerization through chemical reaction of molecular components;
- Thixotropy due to the formation of a structure within the asphalt binder over a long period of time;
- Syneresis due to the exudation of thin oily components; and
- Separation through the removal of oily constituents, resins, and asphaltenes by absorptive aggregates.

Ageing is affected by both internal and external variables. The internal variables refer to the materials and characteristics of a particular asphalt mixture (e.g. bitumen, air void content, and bitumen film thickness) (Kandhal and Chakraborty, 1996). Furthermore, the external variables refer to the production temperature of the mixture (i.e. short-term ageing), and environmental conditions during the service life of the road (Lee, 1973, Traxler, 1963). Excessive hardening can result in a brittle bitumen, with significantly reduced flow capabilities, which contributes to various forms of cracking in the asphalt mixture. Cracking generally occurs in the form of fatigue, thermal or reflective cracking (Scholz, 1995).

The age hardening and cracking potential of binders, can be assessed through the use of rheological parameters that can be determined from measured rheological data. One of the most popular parameters is the Glover-Rowe Parameter. This parameter, which was originally developed by Glover et al. (2005) and then reformulated by Rowe (2011), considers the stiffness and relaxation capability of the binders, and offers an indication of the cracking resistance in asphalt pavements at intermediate temperature.

The $G\text{-}R$ parameter is defined by Equation 21, and it captures the complex shear modulus (G*) and binder phase angle (δ) at a temperature-frequency combination of 15°C-0.005 rad/s. At the frequency of 0.005 rad/s, the G-R parameter gives a boundary value of 0.18 MPa, which is considered as the

onset of cracking, while a value of 0.60 MPa or more is suggested as an indicator of significant cracking (Rowe, 2011).

$$G - R = \frac{G^*(cos^2\delta)}{sin\delta} \tag{21}$$

Since the parameter is calculated at a fixed frequency, it is usually plotted in the Black Space. Figure 2.23, shows a sample data plotted with the Glover-Rowe parameter analysis of recovered binders from mixtures containing RAP and after being aged in the laboratory. The curves in this figure represent the limits of onset and onset cracking. In this plot, it is considered that the materials that exhibit higher $|G^*|$ and lower δ values (moving towards the upper left of the plot) are expected to undergo more age hardening. The Glover-Rowe analysis shows that as RAP material is added, the location in the black space shifts to larger G-R values and lower phase angle values, due to the harder consistency of the bitumen. As the binders are subjected to greater ageing they approach a region in the Black space plot shown as the damage zone. The *G-R* analysis also predicts that two of the mixtures will experience age-induced cracking issues.

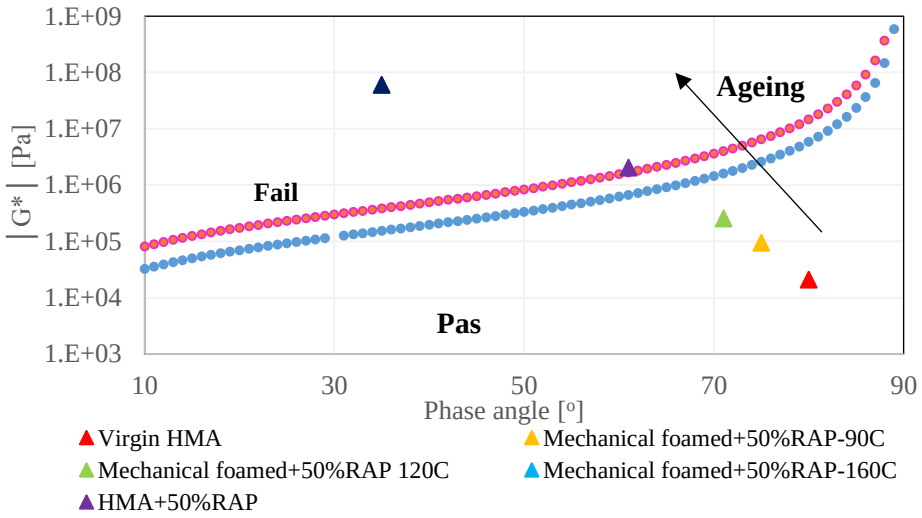


Figure 2.23 Glover-Rowe black space diagram

2.6.3 Approaches to the combined effects of moisture and ageing: Saturating Ageing Tensile Stiffness (SATS) Test

The SATS test was developed in the Nottingham Transportation Engineering Centre (NTEC) at the University of Nottingham in 2005 (Collop et al., 2004, Highways-Agency, 2007), with the objective of analysing the combined effects of ageing and moisture susceptibility in asphalt mixtures. This experimental procedure was originally developed as part of a comprehensive research study to

reproduce in the laboratory the loss of stiffness modulus observed on a trial site located in the UK, where High Modulus Base (HMB) material was placed (Collop et al., 2004, Airey et al., 2005). The data obtained from monitoring the trial segment showed that the stiffness modulus dropped unexpectedly by approximately 60% after eight years. The SATS test was found to successfully reproduce in the laboratory this loss of stiffness modulus (Airey et al. 2005). Furthermore, the SATS test has been shown to correctly predict the performance of asphalt mixtures in the field and replicate the magnitude of moisture damage processes, being able to distinguish between good and poor performing aggregate types (Collop et al., 2004, Airey et al., 2005). In general, the SATS procedure is considered to be more severe than other test procedures to evaluate moisture damage in asphalt mixtures (e.g. the AASHTO T283 procedure) (Airey et al. 2005).

The test consists of subjecting five identical presaturated asphalt mixture cylindrical specimens of 100 mm in diameter and 60 mm in thickness, to a moisture conditioning process at a temperature of 85ºC and a pressure of 2.1 MPa for an extended period of time of 65h. A pressure vessel (partially filled with water) is used to hold the five nominally identical specimens in a custom-made specimen tray as shown in Figure 2.24. P1 to P5 refer to the position of the specimen in the pressure vessel. Two properties obtained from the test are used as an indication of the sensitivity of the compacted mixture to the combined effects of combined ageing and moisture: 1) the stiffness modulus measured after the test divided by the stiffness modulus measured before the test, known as retained stiffness modulus and, 2) the specimen saturation after the test which is known as retained saturation (Airey et al. 2005).

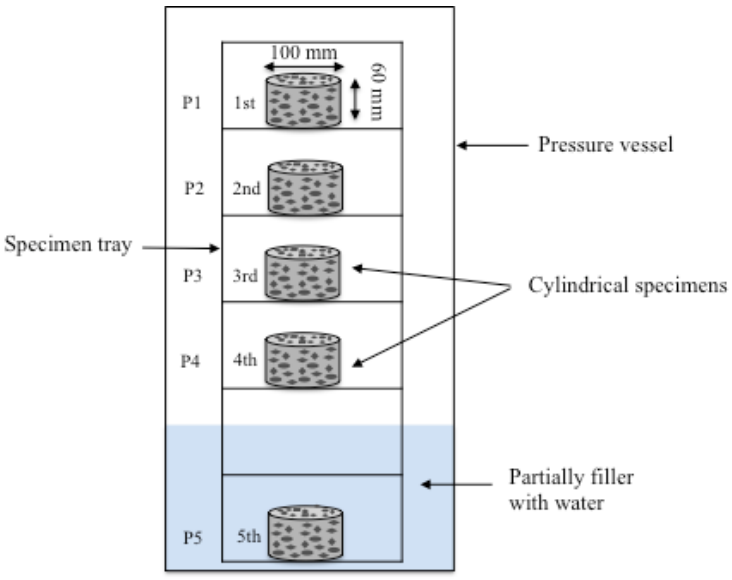


Figure 2.24 Schematic of the specimens in the SATS pressure vessel and specimen tray (After (Airey et al. 2005)

As described in Grenfell et al. (2012), during the test, there is a continuous cycling of moisture within the pressure vessel, which causes condensation on the underside of the top lid and 'dripping' onto the top specimen. There is then a cascading effect where progressively smaller amounts of water 'drip' onto the specimens below, resulting in a decrease in retained saturation level for specimens that are located lower down inside the pressure vessel (Grenfell et al., 2012).

The ten steps of the SATS conditioning and test procedure as specified in Clause 953 are as follows (Grenfell et al. 2012):

1. The unconditioned (initial) indirect tensile stiffness modulus of each asphalt mixture specimen is determined at 20°C using the Nottingham Asphalt Tester (NAT; Cooper and Brown 1989) in accordance with BS EN 12697- 26 Annex C (124 ms rise time, 5μm peak transient horizontal diametral deformation) (BSI 2004a).
2. The dry mass of each specimen is next determined.
3. The specimens are subsequently immersed in distilled water at 20oC and saturated using a residual pressure of 33 kPa (i.e. 68 kPa below atmospheric pressure) for 30 min.
4. The wet mass of each specimen is next determined by weighing, and the percentage saturation of each specimen is calculated, referred to as 'initial saturation'.
5. The SATS pressure vessel is partly filled with a pre-determined amount of distilled water (final water level between the bottom, submerged specimen and the above 'dry' or pre-saturated specimen). The pressure vessel and the water are maintained at the target temperature of 85°C for at least 2 h prior to introducing the specimens.
6. The saturated asphalt specimens are then placed into the pressure vessel, the vessel is sealed and the air pressure is gradually raised to 2.1 MPa.
7. The specimens are maintained at the testing conditions, i.e. 2.1MPa and 85°C for 65 hours.
8. After 65 h, the target vessel temperature is reduced to 30°C and the vessel is left for 24h to cool. When the pressure vessel's display temperature has reduced to 30°C (after the 24h cooling period), the air pressure is gradually released. When the vessel has achieved atmospheric pressure, it is opened and the specimens are removed. Each specimen is then surface dried and weighed in air. The percentage saturation calculated at this stage is referred to as the 'retained saturation' (BSI 2003a, 2004b, 2009).
9. The specimens are finally brought back to 20°C and the conditioned (final) stiffness modulus is determined once more using a NAT.
10. The ratio of the final stiffness modulus to the initial stiffness modulus can thus be calculated, and is referred to as the 'retained stiffness modulus'.

Chapter 3 : FAM mixtures design and experimental set-up

This chapter describes the design of the FAM mixtures, which was used to manufacture all the FAM mixtures evaluated in this work. Furthermore, the DMA and the DSR testing procedures employed for the characterization of the FAM mixtures and binders respectively, are presented.

3.1 Design of FAM mixtures

3.1.1 FAM gradation

The composition of the FAM materials was a derivative of the original HMA mixture, a standard close graded 0/14 mm surface course type asphalt mixture widely used for road construction in the UK. In addition, based on the BS 4987-1:2005 the bitumen content for this asphalt mixture was 5.0% by weight of the total mixture.

Generally, studies that comprise FAM materials use the 1.18mm sieve to separate the coarse and fine aggregate particles (Masad et al., 2008, Caro et al., 2008a, Castelo Branco, 2008). In this study the 1mm sieve was selected, since the 1.18mm does not belong to the British sieve series. The FAM mixtures were then designed following the methodology proposed by Masad et al. (2006a), keeping the same proportions for all the fine particles as in the full mix gradation, normalising their proportions by the percentage passing the largest sieve used (i.e. 1 mm). Figure 3.1 illustrates the gradation of the HMA and the FAM materials.

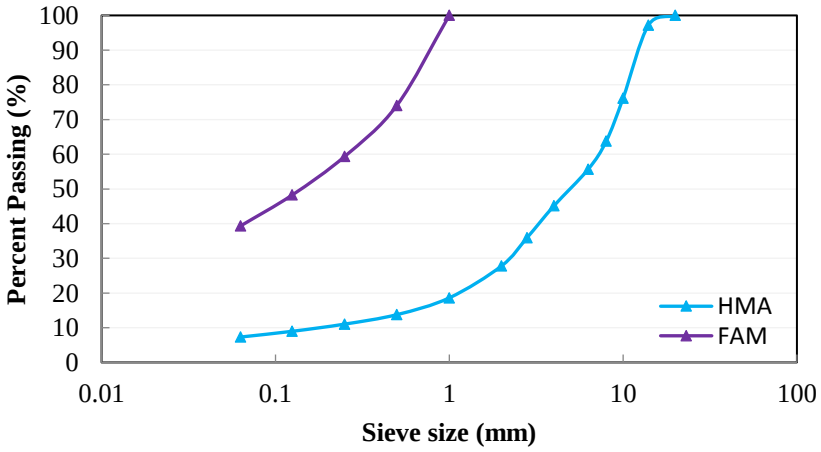


Figure 3.1 HMA and FAM gradation curves

Following this procedure, Table 3-1 shows the FAM gradation, where it can be evidenced that the maximum aggregate size is 0.5mm and the filler content is approximately 39%.

Table 3-1 FAM gradation

Sieve Size (mm)	Percent Passing in HMA (%)	Percent Retained in HMA (%)	Percent Passing in FAM (%)	Percent Retained in FAM (%)
1	18.6	9.2	100	0
0.5	13.8	4.8	74.1	25.9
0.25	11.0	2.7	59.3	14.7
0.125	9.0	2.1	48.3	11.1
0.063	7.3	1.7	39.3	8.9
Bottom	-	7.3	-	39.3

3.1.2 Bitumen content

To determine the bitumen content to be included in the FAM mixtures, a theoretical approach was performed based on the volumetrics of the full mixture. The general procedure consisted of determining the average bitumen film thickness coating each particle, based on the complete gradation of the original full asphalt mixture and bitumen content. Then, this value was used as input for the computation of the bitumen thickness coating the FAM particles, to estimate the amount of bitumen content present in these materials. The procedure followed is listed below:

1. From the HMA used as a reference for the FAM, information regarding the aggregate gradation (percentage passing and percentage retained), and bitumen content was obtained;
2. The mass of each fraction size was calculated based on the total mass of the aggregates in the mixture;
3. The volume of each fraction size was then calculated by dividing the mass of each fraction size determined in step 2 by the bulk specific gravity of the aggregates (Gs≈2.7);
4. The approximate number of particles of each fraction size was determined by dividing the total volume of each fraction size by the volume of one particle within each sieve. In this step, a spherical-shape was assumed for the particles, using the corresponding sieve aperture size for a more accurate reference diameter size for each fraction.
5. The surface area of the particles for each fraction size was determined. In this step, spherical particles were assumed again, using the corresponding sieve aperture size for a more accurate reference diameter size for each fraction, as in step 4.

6. The volume of bitumen for each particle size was calculated by multiplying the surface area calculated in step 5 by the bitumen thickness, which was an unknown value at this point. An initial bitumen thickness of 10 μm was used as a starting point (Kim et al., 2003).
7. The volume of bitumen for all the particles in each fraction size was determined by multiplying the number of particles calculated in step 4 by the volume of bitumen for each particle size determined in step 6.
8. The weight of bitumen for all the particles in each fraction size was determined by multiplying the volume of bitumen for all the particles in each fraction size calculated in step 7 by the specific gravity of the bitumen (Gs≈1.0).
9. The total binder content was determined by adding the weight of bitumen for all the particles in each fraction size determined in step 8. This binder content corresponds to the total binder content present in the original full asphalt mixture, which is a known value (i.e. 5%). Therefore, at this point the average bitumen film thickness coating each particle was changed manually until the desired binder content of 5% was achieved.

After following this procedure, an approximate bitumen film thickness of 11.5 μm was obtained for a 5% binder content, with the full asphalt mixture gradation. This value was considered appropriate since it was found to be similar to values found in the literature (Kim et al., 2003).

Then, this value was used as input to determine the bitumen content present in the FAM materials. This procedure consisted of following the same steps to determine the approximate bitumen film thickness coating the particles in the full mixture described previously, using in this case the FAM gradation that was determined in Section 3.1.1 (Table 1), and the bitumen film thickness determined previously, in step 9. With this procedure, the binder content present in the FAM mixture was found to be 19.5% approximately. This value is not surprisingly high, taking into account the large amount of filler (i.e. 39%) present in the FAM gradation (Table *3-1*). Considering the amount of fine particles within the mixture and the fact that they have higher surface areas, bitumen tends to concentrate in coating these smaller sieve size particles. However, practical considerations should be given to the bitumen content in FAM samples, as it does not always result in testable samples. For instance, very high bitumen contents produce samples that creep under self-weight. Additionally, high filler contents are related to an increase of the stiffness of the mixtures and therefore resulting in fragile specimens. Furthermore, based on the literature regarding FAM materials, (Castelo Branco, 2008, Caro et al., 2008a, De Sousa, 2010, Hernández, 2013, Im et al., 2015) it was found that for different gradations, the bitumen content generally used for these materials is between 8-13% with filler contents varying between 2-27%. These wide ranges are probably associated with the method to estimate the binder content in the FAM materials. However, in general, a ratio of bitumen-filler of 2:1 was observed (Figure 3.2).

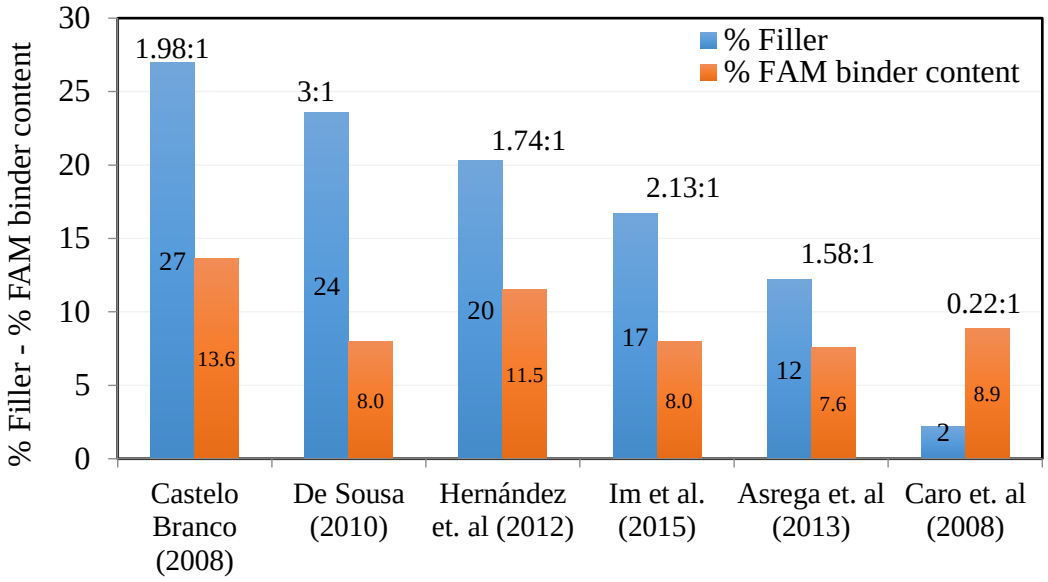


Figure 3.2 Percentage filler and FAM binder content in FAM mixtures based on the literature

Moreover, varying only the filler content (i.e. particles below 0.063mm) in the FAM design obtained in Section 3.1.1, and determining the corresponding bitumen content, higher filler contents resulted in higher FAM binder contents, as shown in Figure 3.3.

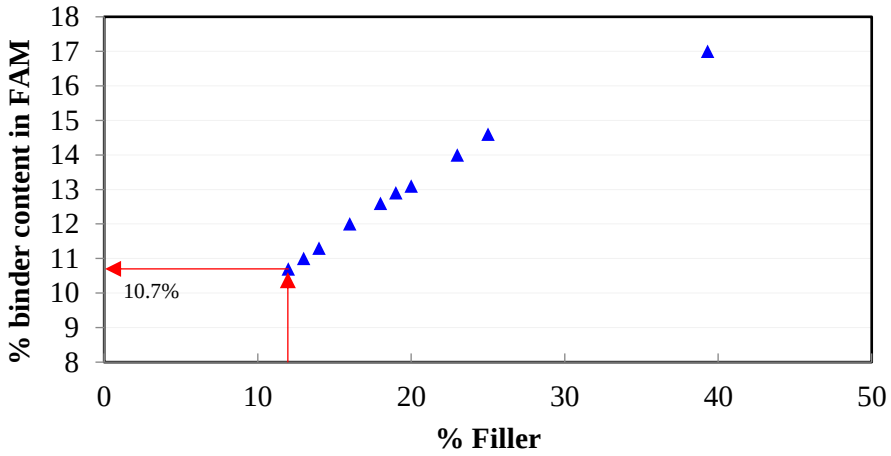


Figure 3.3 Relation between binder content and filler content in FAM

In this regard, and based in all the above-mentioned considerations, the amount of filler in the FAM design was reduced, in order to obtain a FAM mixture with a bitumen content of approximately 10%, which corresponds to the average value used in FAM materials according to the literature. From Figure 3.3, it can be seen that a filler content of approximately 12% resulted in a FAM binder content of 10.7% approximately. Therefore, this filler and bitumen content were selected for the FAM

mixtures, and the proportions of the rest of the fine aggregates were determined by assuming a linear relationship. Figure 3.4 shows the proportioning of the aggregates in the full asphalt mixture and the new modified FAM gradation. In this figure, the initial normalized gradation is presented as well.

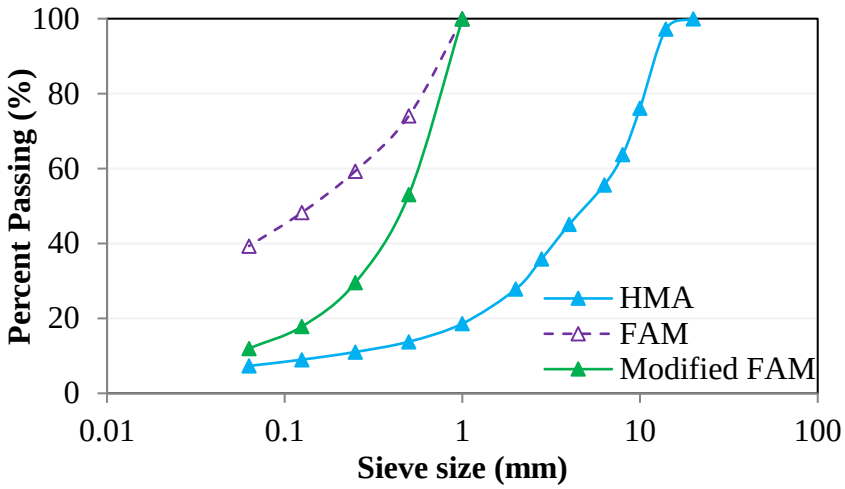


Figure 3.4 Gradation of the full asphalt mixture and the modified FAM

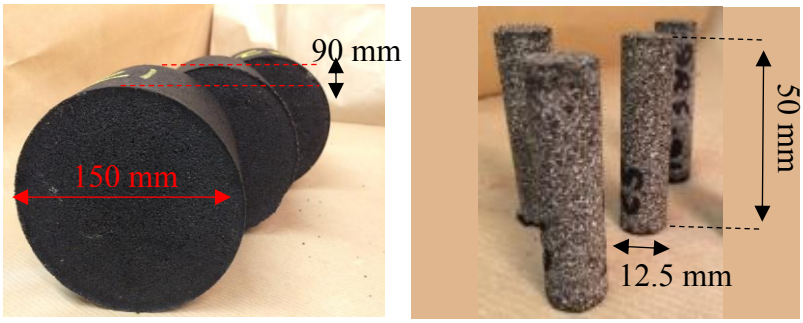
It is noteworthy to mention that the modified FAM gradation is considerable different from the initial FAM design which was a closer representation of the fine matrix existing within the full asphalt mixture. However, these modifications in the FAM design were considered valid for the main focus of the study and therefore this design was used to manufacture all the FAM mixtures evaluated in this work.

3.2 Experimental design

3.2.1 Test procedures to estimate the rheological properties of the FAM materials

A dynamic mechanical analysis (DMA) testing technique was used to determine the rheological properties ($|G^*|$ and δ) of the FAM materials. The general procedure consists of using a rheometer with a special solid fixture configuration to conduct shear controlled strain tests on small cylindrical FAM specimens. DMA rheometers can perform various types of tests depending on the scope of the study. In this case, to obtain the rheological properties of these materials, frequency and temperature sweep tests using the DMA configuration were conducted. The testing protocol for determining the linear viscoelastic properties of FAM materials can be found in Appendix A.

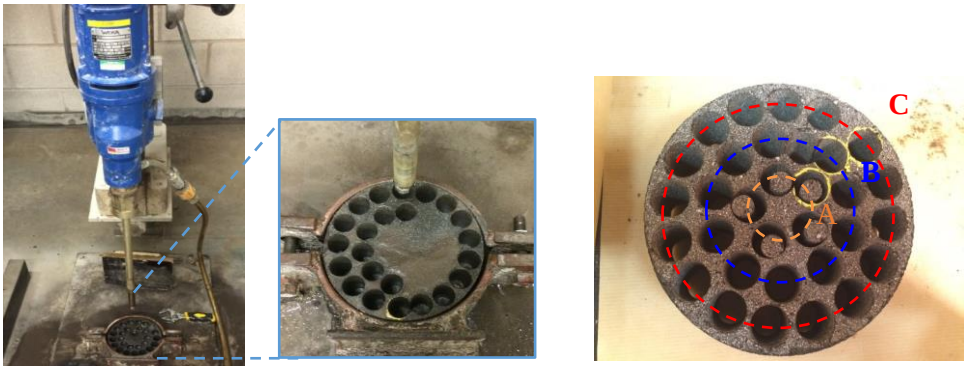
Cylindrical specimens for each FAM mixture evaluated were prepared following the method originally proposed by Masad et al. (2006). In this method, a cylindrical FAM specimen of 150 mm in diameter and 90 mm in height was fabricated using the Superpave gyratory compactor (SGC) (Figure 3.5 a). After compaction, the top and bottom parts of this specimen are trimmed and a coring barrel is used to extract cylindrical samples of 50 mm in height and 12.5 mm in diameter (Figure 3.5 b). For this study, the SGC specimens were compacted following the BS EN 12697-31:2007, targeting a density of 2056 kg/m^3 (i.e. a target air void content of the FAM mixtures of 10%).



(a) (b)

Figure 3.5 Procedure for the fabrication of FAM testing specimens: (a) SGC specimens 150mm in diameter, and, (b) FAM testing specimens' dimensions.

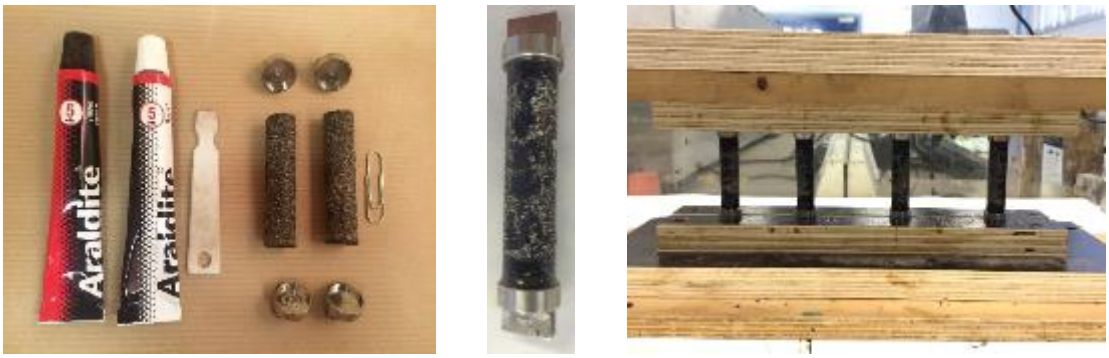
After the small cylindrical specimens were obtained, they were labelled as A, B, or C, where A corresponds to the inner concentric zone of the core, B to the intermediate zone, and C to the outer zone (Figure 6), followed by a number for each sample. For instance a specimen labelled B2, is the second sample that was obtained from the intermediate zone. The air void content for each specimen was determined by its volumetric properties.



a) **b)**

Figure 3.6 coring process of FAM samples for DMA testing (a) coring barrel, (b) labelling

Once the specimens were cored, metallic holders were glued on both ends of each specimen using a two-component epoxy glue (i.e. resin and a hardener) to provide strong adhesion. Once the holders were glued, they were placed in a support to assure that the holders were aligned with the samples, and were left to dry for 24 hours prior to testing. Figure 3.7 illustrates the procedure for specimens' gluing.



(a) (b) (c)

Figure 3.7 Preparation of the FAM specimens for DMA testing (a) epoxy glue and metallic holders, (b) FAM samples with metallic holders on both ends, and (c) drying and vertical alignment of the specimens

These specimens were then used to undertake the DMA testing procedure. A Kinexus DSR rheometer manufactured by Malvern® was used to measure the viscoelastic material properties of the cylindrical FAM specimens. Figure 3.8 illustrates the configuration of the rheometer and a FAM specimen ready to be tested.

(a) (b) (c)

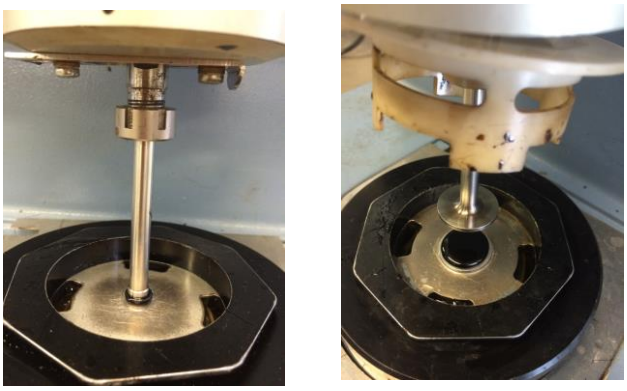
Figure 3.8 DMA testing configuration for FAM materials: (a) FAM sample with metallic holders fixed at the lower geometry, (b) FAM sample with metallic holders fixed at the upper and lower geometries and, (c) Mounting the FAM sample in the rheometer.

The following test conditions were used to obtain the linear viscoelastic properties of all the FAM mixtures evaluated in this study:

- Mode of loading: controlled-strain (of 0.0065%, a value commonly recommended in the literature to test FAM materials in their linear viscoelastic range (Caro et al., 2008, 2015; Castelo Branco, 2008; Masad et al., 2008)).
- Temperatures: 15 to 65^{o}C in 10^{o}C increments
- Frequencies: 0.5‑15.85 Hz (16 readings).

3.2.2 Test procedures to estimate the rheological properties of binders

A Dynamic Shear Rheometer (DSR) machine was used to characterise the viscoelastic properties of the binders evaluated in this study by measuring their rheological parameters over a range of temperatures and frequencies. The instrument set-up comprises two metal plates sandwiching the sample (Figure 3.9), a loading unit connected to the upper plate, an environmental chamber for temperature control, and a data acquisition system. The sample is always sandwiched between the lower plate and the upper spindle all submerged inside a water chamber to reduce any thermal gradient and thermally equilibrate the sample. During the test, the lower plate remains fixed while the upper spindle rotates to implement the oscillatory test for the purpose of this study.



(a) **(b)**

Figure 3.9 Bitumen samples in DSR: (a) 8 mm plates geometry and (b) 25 mm plates geometry

In this study, a Bohlin Gemini 200 DSR machine was used to perform frequency and temperature sweep tests with the following test conditions:

- Mode of loading: controlled-strain (0.6% for 8 mm geometry and 0.8% for 25 mm geometry).

- Temperatures: 5 to 75^{o}C in 5^{o}C intervals
- Frequencies: 0.1 to 10Hz (11 readings), and from 5 to 75^{o}C in 5^{o}C intervals.
- Plate geometries: 8 mm diameter with a 2 mm gap (5 – 45^{o}C), and 25 mm diameter with a gap of 1 mm (25 – 75^{o}C).

Chapter 4 : Rheological properties of FBMs produced with different foaming technologies

This chapter describes the rheological characteristics of FAM mixtures manufactured with two foaming technologies using the same base materials (i.e. bitumen and mineral aggregates) and mixture design. This evaluation includes the effect of production temperature in the final properties of the mixtures by combining the foamed bitumen with aggregates that were previously subjected to different heating conditions or applications: half-warm (i.e. aggregates at 90ºC), warm (i.e. aggregates at 120ºC) and hot (i.e. aggregates at 160ºC) processes. Furthermore, the recovered binders from these mixtures were studied to evaluate the changes in their rheological properties with foaming technology and production temperature of the mixtures.

4.1 Experimental programme

4.1.1 Foaming technologies evaluated

The foaming technologies selected to produce FBMs were: (1) a mechanical foaming technique, and (2) the use of zeolites.

The first technology uses special nozzles to generate the foaming effect by the direct injection of water under high pressure into hot bitumen using a laboratory foaming plant. The amount of water added to generate the foamed bitumen is usually 1% to 5% by weight of the binder. Assuming a foaming water content (FWC) of 3%, this corresponds to 3g of water per 100g of bitumen in the asphalt mixture. For a mixture containing 5% binder content the amount of water will be approximately 0.15%.

The second method includes a regular foamed WMA technology that consists of the addition of zeolites into the bitumen. The dosage rate is typically 5% by weight of the WMA binder. This corresponds to 5g of zeolite per 100g of bitumen, which results in approximately 1.0% water (assuming that the zeolite contains 20% by mass of water, and that all this water is released). For a mixture containing 5% binder content the amount of water will be approximately 0.05%. Therefore, comparing to the mechanical process to produce foamed bitumen mixtures (where there is

approximately 3g of water per 100g of bitumen in the mixture), the foamed bitumen mixtures by the mechanical process contain 3 times more water than the zeolite foaming technologies.

Details on the FBMs production with these technologies are explained in Section 4.1.4.

4.1.2 Material selection

For this study a conventional 70/100 penetration grade bitumen was selected as it is widely utilised for asphalt pavement construction in the UK. According to BS 2000-49:2007 and BS 2000-58:2007, the penetration grade at 25ºC and softening point were found to be 73 dmm and 46ºC respectively. Moreover, the mineral aggregates used were limestone. This type of aggregates was selected because of its frequent use in asphalt paving mixtures. They primarily consist of calcium carbonate and are considered chemically stable with little reaction with bitumen (Liao, 2007).

4.1.3 Characteristics of the FAM mixtures

Nine FAM-FBMs were manufactured in the laboratory using the FAM mixture design presented in Section 3.1 and using the same base materials (i.e. bitumen and fine aggregates) described previously. These mixtures include:

- Three FAM mixtures - no foam bitumen technology- (Standard-FAM),
- Three FBMs produced through a laboratory foaming plant (Mechanical Foamed-FAM), and
- Three FBMs containing zeolites (Zeolite-FAM).

Three different mixing temperatures (i.e. 90, 120 160ºC) were employed for the different mixtures. For the Mechanical Foamed-FAM mixtures, the bitumen was heated at 160ºC in all cases for foaming purposes, and the aggregates were heated 20ºC above the corresponding mixing temperatures in order to achieve the targeted mixing temperatures (i.e. 90, 120 160ºC). For the Zeolite-FAM mixtures and for the Standard-FAM mixtures as well, both bitumen and aggregates were heated at 90, 120 160ºC to achieve these target final temperatures of the mixtures. Table 4-1 shows the temperatures of the constituent materials and the final target temperature of the mixtures.

Table 4-1 Characteristics of the FAM mixtures

Mixture Type	T of the Materials (ºC)		Mixing T (ºC)	Foaming technology
	Bitumen T (ºC)	Aggregates T (ºC)		
Standard-FAM-90C	90	90	90	None
Standard-FAM-120C	120	120	120	
Standard-FAM-160C	160	160	160	
Mechanical foamed-FAM-90C	160	110	90	Mechanical foaming
Mechanical foamed-FAM-120C	160	140	120	
Mechanical foamed-FAM-160C	160	180	160	
Zeolite-FAM-90C	90	90	90	Zeolite
Zeolite-FAM-120C	120	120	120	
Zeolite-FAM-160C	160	160	160	

Regarding the mix design procedures, following previous research on WMA design practices (which includes the zeolite foaming technologies) (Bonaquist, 2011, West et al., 2013) the WMA design has been based on the HMA mixture design with no change in the job formula, in terms of the materials selection (i.e. bitumen and aggregates). Furthermore, in terms of the foamed bitumen mixtures manufactured by the mechanical process, there is no standardised method to design foamed bitumen mixtures with this technology. Most of the agencies which use FBMs (Academy, 2009, Wirtgen, 2010) have their own mix design procedures which are the result of numerous efforts over decades (Jenkins, 2000). Several research efforts have been conducted to identify the critical mix design parameters in this type of mixtures (e.g. (Sunarjono, 2008)). However, despite these efforts, foamed bitumen application in warm processes does not follow a standardised mix design procedure. Consequently, the mix design parameters required for these mixtures such as foam characteristics, mixing, compaction, curing and testing are far from being standardised (Kuna et al., 2014). Therefore, for this study the three groups of FAM mixtures were manufactured using the same base materials, the same volumetric properties and the same base design used for a standard HMA mixture described previously.

Cylindrical FAM specimens of 50 mm in height by 12.5 mm in diameter were prepared for each FAM mixture following the methodology described in Section 3.2. These specimens were used to undertake the DMA testing procedure. For the purpose of this application, frequency and temperature sweep tests were conducted on the cylindrical fine Standard-FAM, Mechanical foamed-FAM and Zeolite-FAM specimens to determine the linear viscoelastic material properties using the testing conditions described in Section 3.2.1. Two specimens were tested after observing low variability in their

rheological properties. The difference between the $|G^*|$ values was below 10%, hence, testing a third specimen was deemed unnecessary. Since the SGC process results in different air void distribution along the sample, the resultant cylindrical samples contain different air void contents as well. Therefore, the specimens to be tested were carefully selected to have similar air void contents to be able to compare the impact of the components of the mixtures in their mechanical properties Figure 4.1 presents the measured air void content determined by volumetric calculations for the tested specimens for the nine mixtures evaluated.

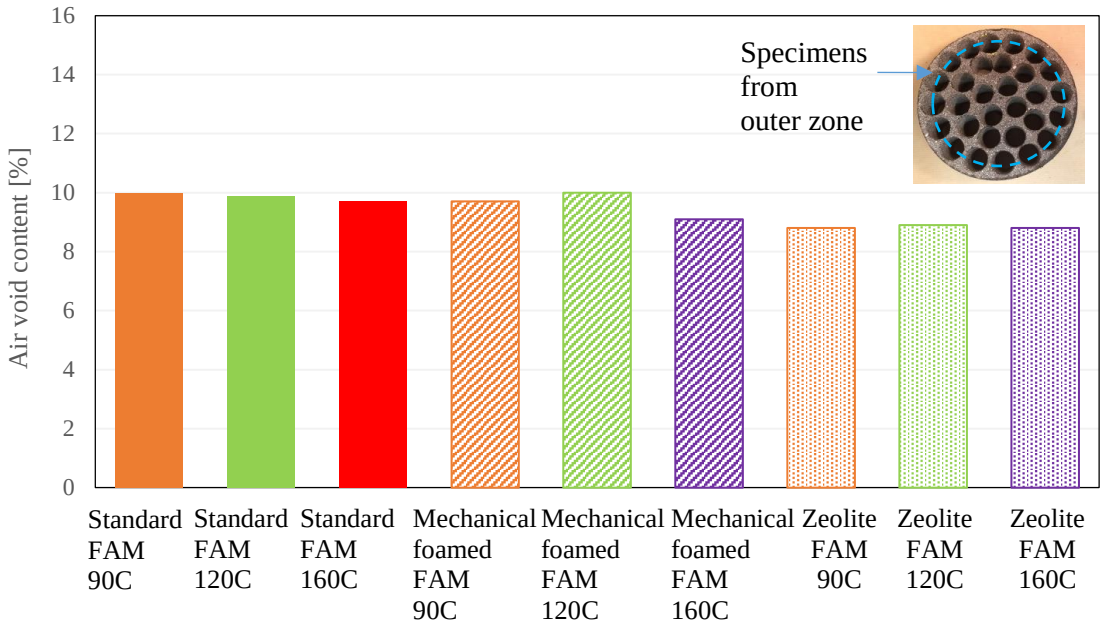


Figure 4.1 Air void contents of all these FAM DMA specimens

4.1.4 FBMs production

For the FBMs produced by the mechanical process, a laboratory mobile foaming plant type Wirtgen WLB 10 S, developed in 2008 was used (Figure 4.2). This mobile plant is specially designed to produce foamed bitumen under laboratory conditions, to evaluate the foaming characteristics of the bitumen and to generate foamed bitumen mixtures as well by attaching a mechanical mixer to the foaming unit (Sunarjono, 2008).

This foaming unit is designed to regulate the amount of dispensed bitumen and water by mass flow meters. The bitumen is heated at high temperatures, which are usually between 160 and 180°C, and circulated inside the unit. Then, the foamed bitumen is produced by combining specific quantities of water, compressed air, and heated bitumen inside an expansion chamber. After the bitumen is foamed, it is usually dispensed directly from the nozzle into the mixer, where it is combined with the aggregates (Newcomb et al., 2015).

The mixer type WLM 30 in this laboratory asphalt plant is designed mainly to produce FBMs in combination with cold, moist aggregates. Therefore, in order to produce FBMs incorporating a certain amount of heat into the aggregates, mixing was carried out using a Hobart type mixer with a flat type agitator (Figure 4.2), which has been used in previous studies to manufacture foamed bitumen mixtures in the laboratory (Sunarjono, 2008). The advantage of this mixer type is that it allows the aggregates temperature to be maintained during mixing. Ideally, a twin shaft pugmill mixer is preferred to manufacture these mixtures, since it produces mixtures that closely represent those produced in field (Long et al., 2004). However, this mixer was not available in the NTEC laboratory to manufacture foamed bitumen mixtures with heated aggregates.

(a) (b)

Figure 4.2 (a) Laboratory foaming plant (b) Hobart type mixer with flat type agitator

During the mixing process and while the squirt of foamed bitumen comes out from the nozzle, the paddle is continuously spinning. This dynamic process aides to achieve better coating of the aggregates while taking advantage of the increased volume of the bitumen while in its foamed state, which lasts only a few seconds. Sufficient amount of mixture is added to the Hobart mixer bowl to ensure that there is enough material to fill the bowl and reach the bitumen.

Aggregates were heated up 20^{o}C above the corresponding final mixing temperature of the mixtures (Table 2) for 6h prior to mixing. Then, these previously heated aggregates were added in the mixer bowl, which was previously heated as well, and were mixed for about 30 seconds to achieve a homogeneous mix. After 30 seconds, and while the aggregate mixing was still in progress, foamed bitumen was sprayed from the nozzle into the bowl and directly mixed with the pre-heated aggregates for up to 3 minutes until all the aggregates were fully coated. The bitumen foaming temperature was 160^{o}C in all cases, and a foaming water content of 3% by mass of asphalt binder was used. The FWC was selected based on previous studies performed in NTEC using similar materials and mixture

design (Kuna et al., 2014). The abovementioned foaming conditions were used for all the mixtures manufactured with this technology.

For the FBMs produced by the addition of zeolites, foamed bitumen was produced using Advera® synthetic zeolite produced by PQ Corporation. This additive comes in a finely powdered (passing sieve No. 200) form, and according to the producer it contains 20% of water in its internal structure (PQ Corporation, 2012). The manufacturing process with this technology consists of pre-blending the zeolite (which was added at room temperature) with the bitumen, which was pre-heated at the corresponding mixing temperature (i.e. 90-160ºC), for further incorporation into the heated aggregates. This technology is convenient to use in the laboratory for mixing purposes, since the zeolite is added in finely powdered form and there is no need for special equipment.

For the preparation of the FAM mixture with this technology, initially the aggregates were removed from the oven (i.e. aggregates where heated at the corresponding mixing temperature of the mixtures for 6h according to BS EN12697-35:2004) and added to the mixer, where they were mixed for 30 seconds. The zeolite was pre-blended with the bitumen, which was heated at the corresponding mixing temperature for 3-5 hours following the BS EN12697-35:2004, and then this blend was added straight to the heated aggregates. Hand mixing was then performed until the aggregates were fully coated. The dosage rate was determined based on the manufacturer's recommendation corresponding to 5% of zeolite by weight of the bitumen. During the manufacturing process of these mixtures a different foaming reaction of the bitumen was observed when Advera® was in contact with the bitumen heated at different temperatures (90-150ºC). Therefore, this foaming action with temperature was studied in more detail.

Three Advera®-bitumen blends were prepared by manually mixing the Advera® powder (added at room temperature) with the bitumen (which was previously heated at 90, 120 and 150ºC), in small tins (Figure 4.3). The 70/100 penetration grade bitumen described in Section 4.1.2 was utilised. It was observed that at lower bitumen temperatures, more bubbles appeared on the surface. This foaming effect with temperature could be explained by changes in the viscosity of the bitumen at the different temperatures. At 150ºC, the bitumen has low viscosity (i.e. behaving more like a fluid) so the air trapped to form the bubbles could be released easily. At 120ºC the viscosity of the bitumen is still low so some of the air trapped in the bubbles can still be released. Moreover, at 90ºC the bitumen has high viscosity (i.e. behaving more like a solid) making it harder for the air in the bubbles to be released.

(a) (b) (c)

Figure 4.3 Advera®-bitumen blends with a 70/100 penetration grade bitumen with bitumen at: (a) 150ºC, (b) 120ºC and, (c) 90ºC

Another set of Advera®–bitumen blends were prepared with a harder penetration grade bitumen (i.e. 10/20 pen) at the same mixing temperatures (i.e. 90ºC, 120ºC and 150ºC) to further evaluate the behaviour. These blends are shown in Figure 4.4. With this harder penetration grade bitumen, some bubbles were noticed at 150ºC (with the 70/100 pen grade bitumen no bubbles were observed at this temperature), and at a lower temperature of 120ºC more vigorous bubbling was observed (some bubbles were observed at this temperature with the 70/100 pen grade bitumen). At 90ºC, mixing was not possible due to the high viscosity of the bitumen at this low temperature. This behaviour did not appear to affect the overall performance of the mixtures being produced with the zeolite, as described in the next section; however, these observations suggest that the release of water from the zeolite depends on the properties of the bitumen (i.e. on bitumen viscosity, mainly).

(a) (b) (c)

Figure 4.4 Advera®-bitumen blends with a 10/20 penetration grade bitumen with bitumen at: (a) 150ºC, (b) 120ºC and, (c) 90ºC

Furthermore, in order to evaluate the rate at which Advera® releases the water with temperature (without the presence of bitumen), 100g of Advera® were placed in the oven at the three evaluated temperatures (i.e. 90ºC, 120ºC and 150ºC). The samples of Advera® were weighed every 10 minutes initially, and a slow decrease in the remaining mass was observed. After more than one hour, still a slow decrease in its weight was observed and this tendency remained even after 6 hours. After this

time, for the Advera® at 90ºC, the mass loss was only 4%. Moreover, for the Advera® at 120ºC and 150ºC, the mass loss was 7 and 9% respectively (Figure 4.5). These results suggest that the water release in Advera® is a slow process that extends over a long period of time, which in fact could improve some mixture properties (i.e. workability) particularly at reduced temperatures.

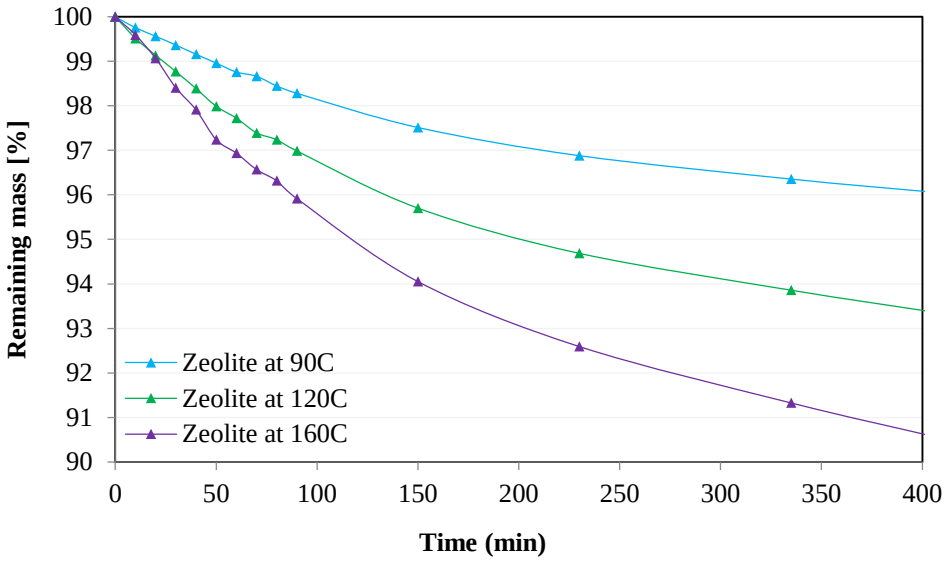
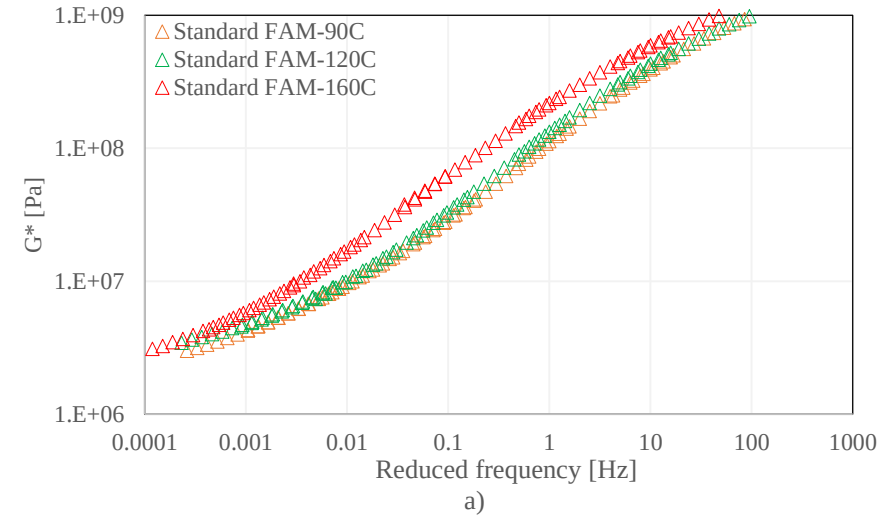


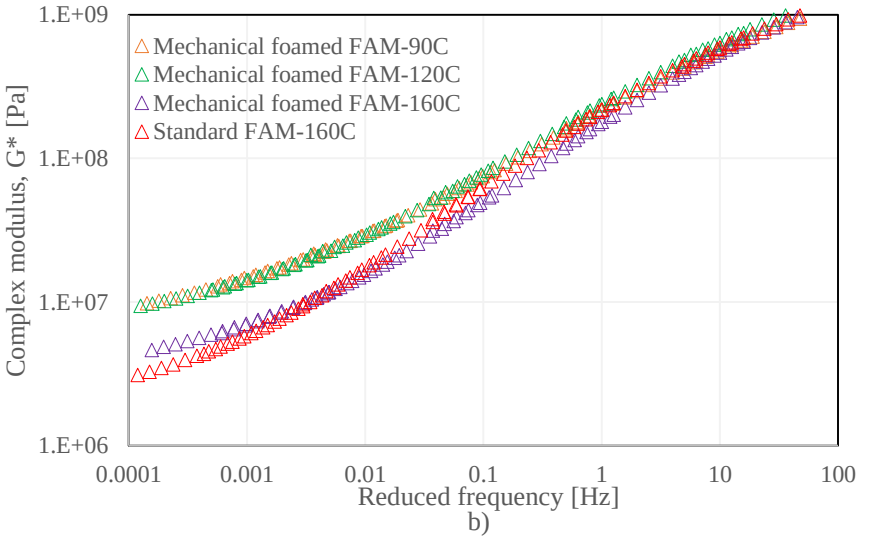
Figure 4.5 Water release in the Advera® Zeolite

4.2 Dynamic Mechanical Analysis results

4.2.1 Dynamic Shear Modulus Master Curves

Figure 4.6 shows the average $|G^*|$ master curves for the three FAM mixtures groups at a reference temperature of 25ºC. In the FBMs figures, the Standard-FAM mixture produced at 160ºC is also presented as a reference for the control mixture for comparison purposes. These curves correspond to the average of the tested specimens for each type of mixture.





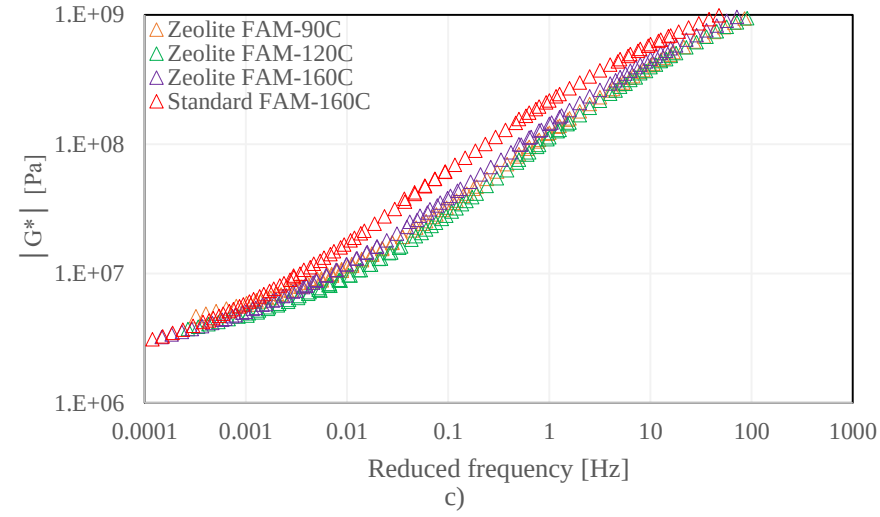


Figure 4.6 $|G^*|$ master curve for: a) Standard-FAM mixtures, b) Mechanical foamed-FAM mixtures, and c) Zeolite-FAM mixtures, at a reference temperature of 25ºC

From these master curves, two initial observations can be made: 1) the upper part of the master curves for all three types of mixtures approaches asymptotically to a unique maximum stiffness of the mixes, which is higher than $1x10^9$ Pa for the considered mix design. 2) At lower frequencies (or higher testing temperatures), the modulus approaches a minimum value, which is different for the two foaming FAM groups. For instance, for the Zeolite-FAM mixtures this lower limiting value is very similar at all production temperatures, and also to that of the Standard-FAM mixtures. However, for the Mechanical foamed-FAM mixtures, the minimum stiffness value is different for all production temperatures, and also different to the value reported for the Standard-FAM-160°C. These differences in the lower limiting values for the Mechanical foamed-FAM mixtures are potentially a consequence of the effects of the foaming technology in the final properties of these mixtures.

With the objective of better quantifying the actual effect of the production temperature and foaming technology on the $|G^*|$ of the FAM mixtures, Figure 4.7 presents the values of $|G^*|$ for all the mixtures at two loading frequencies (0.001Hz and 10Hz), at the reference temperature (25°C).

Data in Figure 4.7 reflects the impact of the production temperature on the changes in the $|G^*|$ of the FAM mixtures manufactured with different technologies, and these changes depend on the loading frequency (Figure 4.7a vs Figure 4.7b). At 10Hz, the Standard-FAM mixtures show an increase in $|G^*|$ values with increasing production temperature. The $|G^*|$ of the Standard FAM mixture manufactured at 120 and 160°C increased by 11 and 51% respectively, with respect to the Standard FAM mixture manufactured at 90°C. This increase in the $|G^*|$ values with production temperature can be attributed to better coating of the aggregates with the bitumen at higher production temperatures, which improved the overall adhesive properties of the mixtures and therefore resulted in better mechanical capacity. For the Mechanical foamed-FAM mixtures, data in Figure 4.7a shows that these mixtures do not follow a constant increasing trend in $|G^*|$ with production temperature. The changes of the $|G^*|$ values in these mixtures are less than 12%, suggesting that there is no effect of temperature for these mixtures. The Zeolite-FAM mixtures, show similar $|G^*|$ values at production temperatures of 90 and 120°C (i.e. changes in $|G^*|$ less than 5%), and a slightly higher increase in $|G^*|$ at 160°C (i.e. $|G^*|$ of the Zeolite-FAM 160C increased by 13% with respect to that of the Zeolite-FAM 90C). These changes in $|G^*|$ are small, suggesting that the effect of production temperature on these mixtures is not very significant.

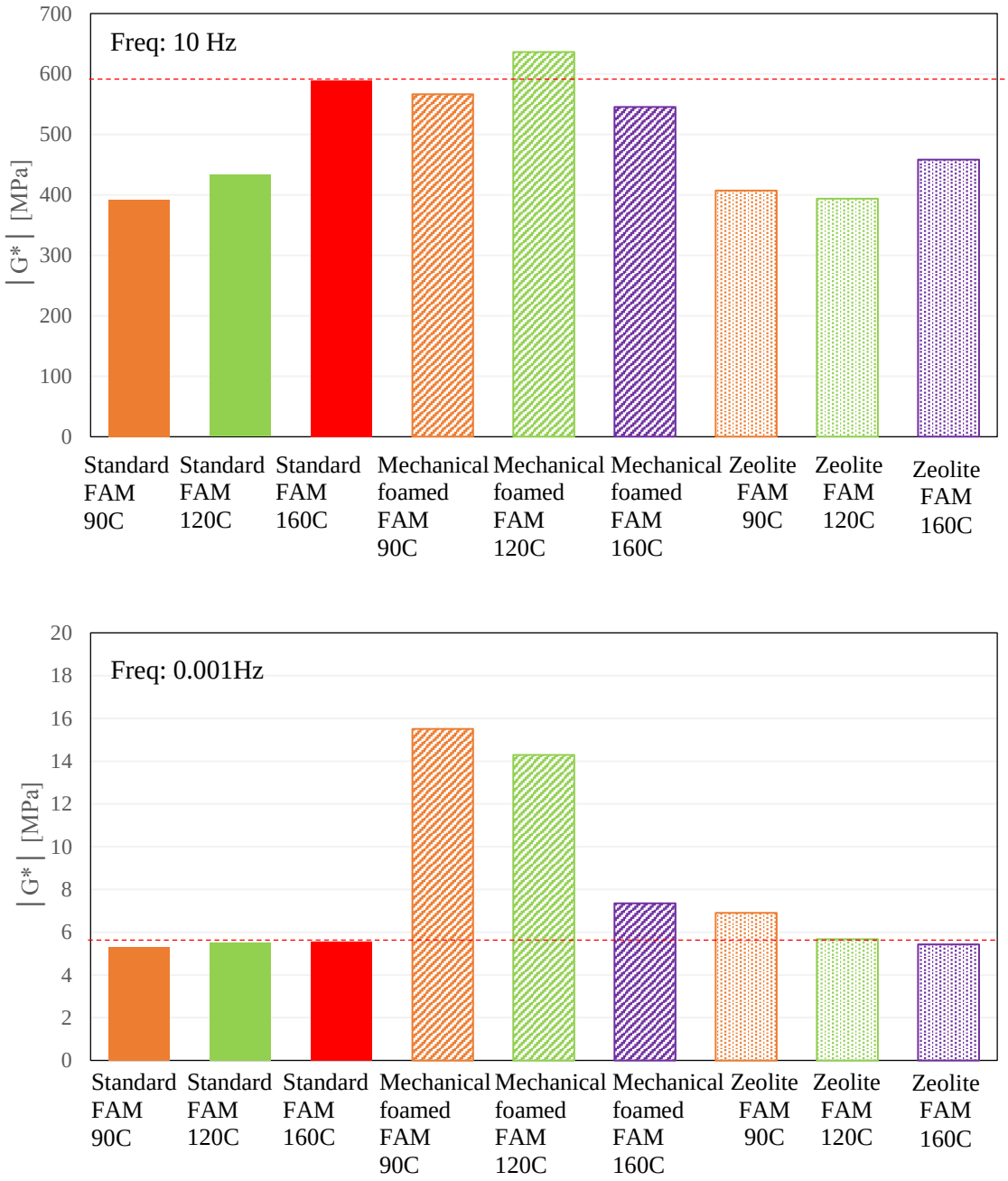


Figure 4.7 $|G^*|$ for all mixtures at (a) 10Hz, and (b) 0.001Hz

Figure 4.7b confirms that the $|G^*|$ values are smaller at lower reduced frequencies (or higher temperatures). At 0.001Hz, the values of the dynamic modulus are within one or two orders of magnitude lower than those obtained at 10Hz. Furthermore, the changes in $|G^*|$ for each type of mixture with production temperature are different compared to those observed previously at higher frequencies. At 0.001Hz, the Standard-FAM mixtures exhibit similar $|G^*|$ values, with slight increase with production temperature (i.e. increases less than 5%). The Mechanical-foamed FAM mixtures on the contrary, show a clear increase in $|G^*|$ with lower production temperatures. For instance, the $|G^*|$ of the Mechanical foamed FAM mixtures manufactured at 90 and 120°C, is

respectively 4.2 and 3.2 times higher than the $|G^*|$ of the Mechanical foamed FAM mixture manufactured at 160ºC. This increased stiffness at low production temperatures for the Mechanical foamed-FAM mixtures appears to be unusual as it was expected that the incorporation of a certain amount of heat to the aggregates for further mixing with the foamed bitumen (which was always heated at 160ºC for foaming) would result in FBMs with better mechanical capacity, reflected in an increase in the $|G^*|$ values. However, this unusual increase in stiffness at lower production temperatures could be attributed to the mixing process employed for the production of these mixtures. For instance, it is possible that when the foamed bitumen is squirted, it only reaches the portion of aggregates that are on the top, and at low mixing temperatures (i.e. 90 and 120ºC) complete dispersion of the bitumen into the aggregates is not fully achieved. In addition, a portion of the added bitumen remains in the mixer and on the vertical agitator after mixing inducing bitumen loss. These two conditions could result in mixtures with different effective binder contents (which explains the difference in the lower limit stiffness values with respect to the Mechanical foamed FAM-160C and also to the Standard FAM-160C). In addition, at low frequencies, where the increase in $|G^*|$ is more evident with lower production temperatures, the lower effective binder content along with the reduction of the viscosity of the bitumen, allow the fine aggregate skeleton to control the rheological properties of the Mechanical foamed-FAM mixtures, providing an increase in $|G^*|$.

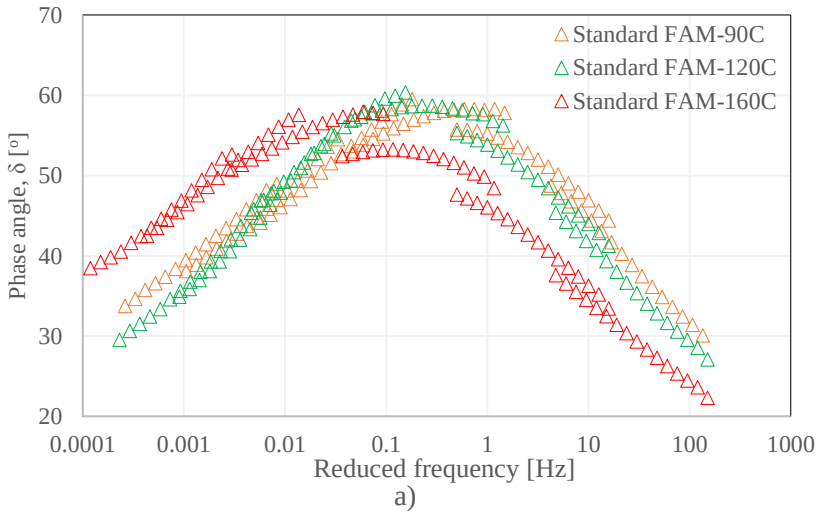
Furthermore, with regards to the Mechanical foamed mixture manufactured at 160ºC, results from both frequencies indicate that producing foamed bitumen mixtures by means of a Mechanical process with a mixing temperature of 160ºC (i.e. aggregates at 160ºC), results in a mixture with similar properties to that of a standard HMA, as could be expected. Considering that the main purpose of the foamed bitumen is to provide adequate workability and coatability conditions to produce mixtures for cold mix processes (i.e. non-heated aggregates) or half-warm mix processes (i.e. partially heated-aggregates) which otherwise could not be achieved with the same bitumen in liquid state, obtaining also environmental benefits. These results imply that extending the production of FBMs for hot processes is not practical, since a mixture with adequate coating and workability conditions promising good mechanical performance at these high temperatures (i.e. HMA) can already be obtained without the need to include special equipment to produce the foamed bitumen, and in addition the benefits for reduced energy consumption within the sustainability framework are no longer effective.

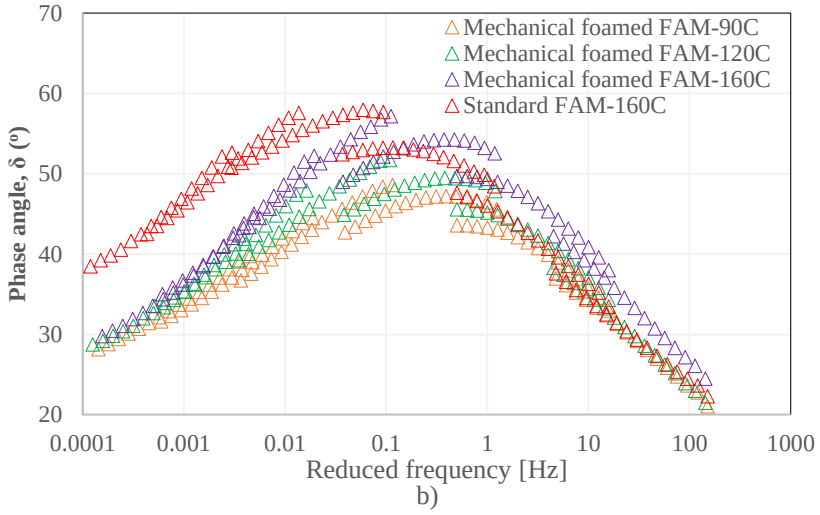
Furthermore, at 0.001Hz, the Zeolite-FAM mixtures exhibited changes in the $|G^*|$ values of less than 20%, for all production temperatures. Although the changes in $|G^*|$ for the zeolite containing mixtures are higher than those exhibited at 10Hz (Figure 4.7a), the production temperature still did not result in major differences in the rheological properties of these mixtures. In addition, another important observation for these type of mixtures which is also reflected in Figure 4.7, is that these

mixtures exhibit comparable $|G^*|$ values to those of the Standard FAM mixtures at the same corresponding production temperature. On average the Zeolite FAM mixtures exhibited changes in $|G^*|$ between 3 to 20%, compared to those of the corresponding Standard-FAM mixtures. These results suggest that the addition of zeolites in the FAM mixtures did not present significant changes in the rheological properties of the final mixtures. It could be possible that during the process of pre-blending Advera® with the bitumen prior to its incorporation into the hot aggregates, the influence of water and foaming process will have disappeared before mixing started making this procedure very similar to just mixing and compacting conventional HMA at lower temperatures. The Mechanical foamed FAM mixtures, on the contrary, exhibit higher changes in the $|G^*|$ compared to those of the Standard FAM mixture at the same corresponding production temperature. On average, the Mechanical foamed FAM mixtures manufactured at 90 and 120ºC exhibited changes in $|G^*|$ between 44 to 191%, compared to those of the Standard-FAM mixtures at the same corresponding production temperatures of 90 and 120ºC. At a production temperature of 160ºC, the $|G^*|$ of the Mechanical foamed FAM mixtures exhibit changes up to 30%, compared to those of the Standard FAM-160ºC. These results suggest that for these types of mixtures, the mixing processes are critical in the rheological response of the materials. In addition, the wide range of changes in $|G^*|$ as a function of the foaming technology suggest that the changes in the rheological properties of the mixtures are highly dependent on the production process.

4.2.2 Phase Angle Master Curves

Phase angle master curves for the three FAM groups were produced by using the shift factors derived from the $|G^*|$ master curves, and they are presented in Figure 4.8 for the three FAM groups. However, unlike the $|G^*|$ master curves where the rheological data produced a smooth continuous curve in all the FAM groups at all production temperatures of the mixtures, the resulting phase angle master curves for all the FAM groups after the shifting procedure resulted in non-continuous, branching and spread waves at all production temperatures of the mixtures. This fact does not allow the TTSP to be applied for the FAM mixtures. However, the experimental rheological data at all the tested temperatures can still be shifted to the reference temperature with respect to time, applying the Partial Time-Temperature Superposition Principle (PTTSP) to produce the δ master curves, and these curves are presented in Figure 4.8.





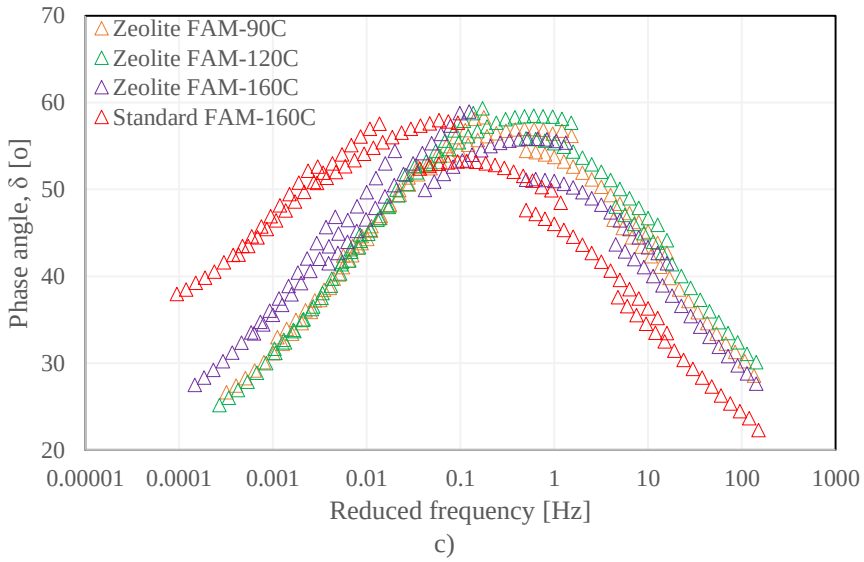


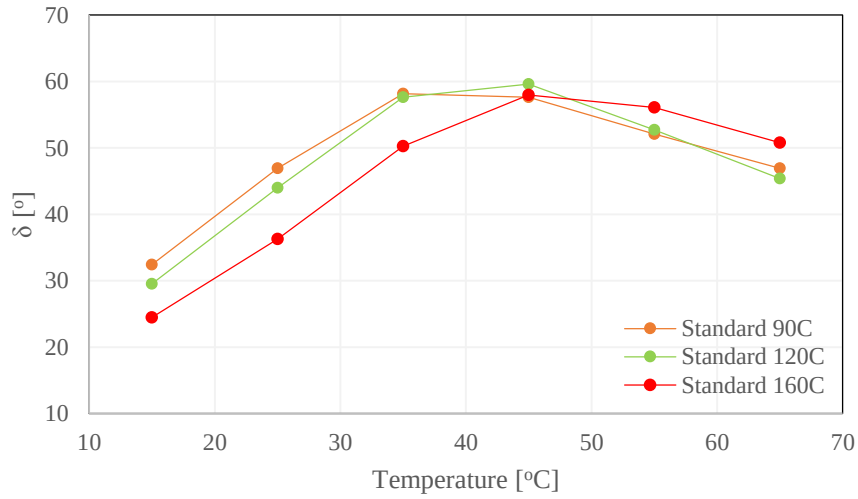
Figure 4.8 δ master curve for: a) Standard-FAM mixtures, b) Mechanical foamed-FAM mixtures, and c) Zeolite-FAM mixtures, at a reference temperature of 25ºC

From these figures, it is possible to observe that the production temperature and foaming technology have an effect on the phase angle of the materials, and as in the $|G^*|$, this effect is a function of the loading frequency. For instance, the δ master curves for the Standard-FAM mixtures in Figure 4.8.a, show that at high reduced frequencies (low temperatures) these mixtures exhibit lower values of δ (more elastic behaviour) with production temperature. This means that the production temperature, affects the viscous component of the bitumen present in the FAM mixtures, leading to stiffer mixtures, as was seen by an increase in the $|G^*|$ master curves, and less viscous material (lower δ values) with production temperature. This behaviour implies that low production temperatures of 90 and 120ºC could be potentially good for a better fatigue resistance of the mixtures. At low reduced frequencies (high temperatures) instead, the Standard-FAM mixtures manufactured at 90 and 120ºC displayed similar values, and the Standard FAM mixtures manufactured at 160ºC exhibited higher δ values. This behaviour (i.e. more viscous response with production temperature) could be an effect of the frequency on the material.

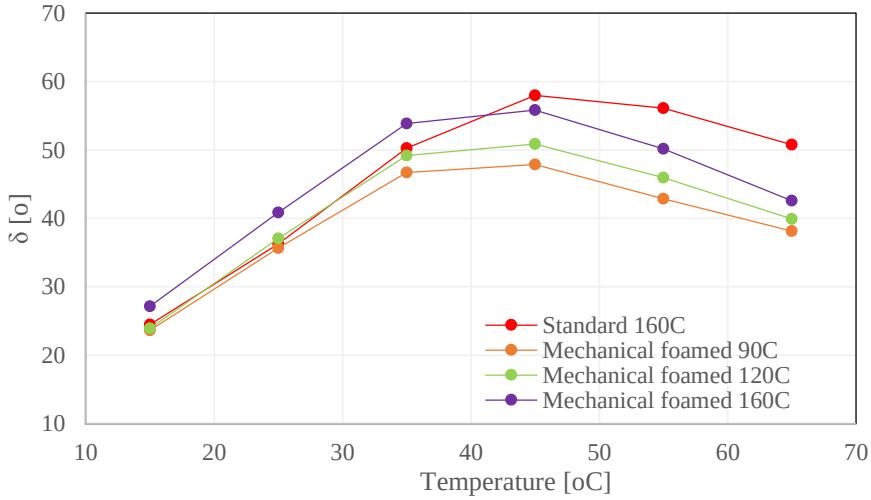
The phase angle master curves for the Mechanical foamed FAM mixtures (Figure 4.8.b), on the contrary, show slightly smaller phase angle values at lower production temperatures over the reduced frequency range. This move towards a more elastic response at lower production temperatures, is an effect of the increased stiffness observed in the $|G^*|$ master curves of these mixtures at production temperatures of 90 and 120ºC. The phase angle master curves of the Zeolite FAM mixtures in Figure 4.8.c exhibit similar phase angle values within the mixtures across the reduced frequencies range, meaning that for these mixtures, the production temperature did not have an effect on the rheological properties of the mixtures, as was observed also in their $|G^*|$ master curves. These results imply that the zeolite containing mixtures are not very sensitive to changes in the mixing temperature of the materials.

Furthermore, comparing the phase angle master curves for the two foaming FAM groups with that of the reference Standard-FAM 160C mixture, both foaming FAM groups exhibit a more elastic response at all production temperatures (lower δ values), at the low frequencies, which corresponds to high temperatures. At the high-reduced frequency end (lower temperatures) the phase angle values are similar.

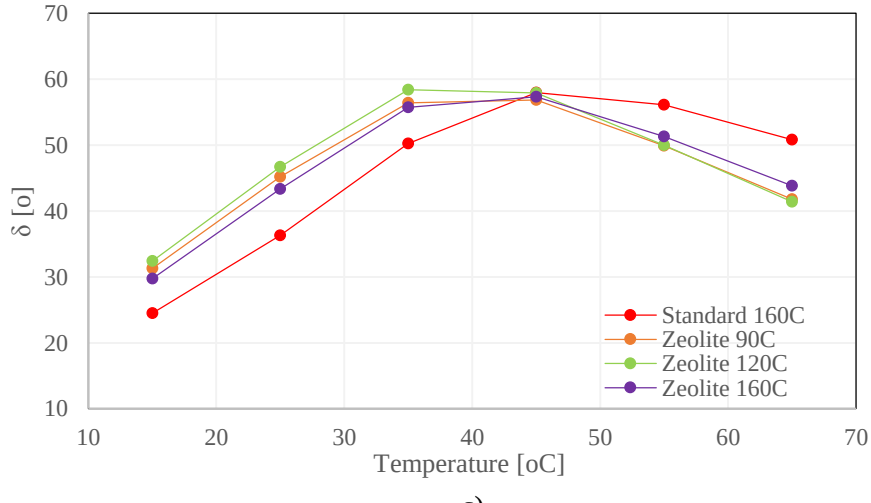
Due to the complex behaviour of these FAM materials, the effects of production temperature and foaming technology with regards to their phase angle were also evaluated by means of the phase angle isochrones. In these plots, the phase angle data obtained from the DMA test, versus temperature at a constant frequency is used, without the need to perform TTSP manipulations of the raw data. Figure 4.9 presents the isochronal plots for the three FAM groups at 10Hz, which is a frequency commonly used in the design of pavements.



a)



b)



c)

Figure 4.9 Isochronal plots at 10Hz for: a) Standard-FAM mixtures, b) Mechanical foamed-FAM mixtures, and c) Zeolite-FAM mixtures, at a reference temperature of 25ºC

The isochronal plots for all three FAM groups illustrate that similar to what has been observed in the δ master curves, the mixtures exhibit a maximum δ value which occurs at 35 or 45ºC, after which there is a decrease in the δ values towards a more elastic behaviour as the temperature increases, and the behaviour of the bitumen is not rheologically dominant.

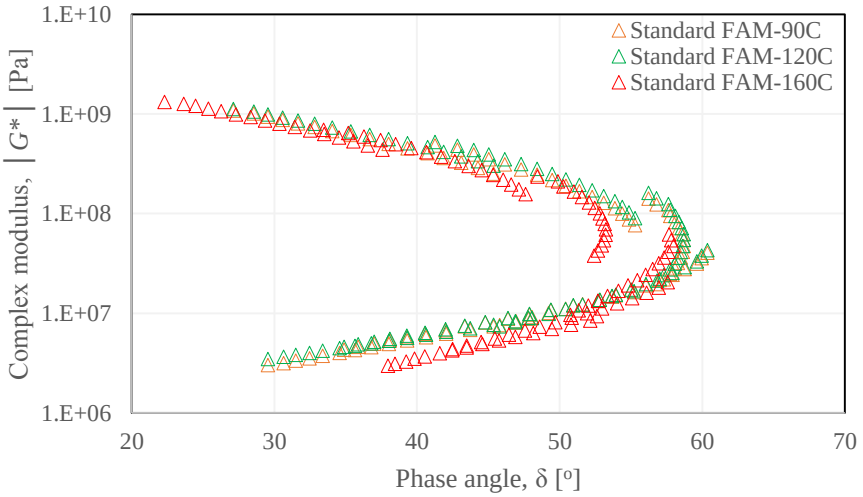
In terms of the production temperature, the isochronal plots for the Standard FAM mixtures in Figure 4.9a, illustrate that there is a decrease in δ with production temperature from 15 to 45ºC, after which there is a sharp increase in the slope of the δ isochrones for the Standard FAM mixture manufactured at 160ºC. The isochronal plots for the Mechanical foamed FAM mixtures (in Figure 4.9b) on the contrary, show a decrease in δ with lower production temperatures, which is maintained throughout the temperature range of 15 and 65ºC. This reduction in δ is associated with the increased stiffness observed in the $|G^*|$ master curves described previously. The isochronal plots for the Zeolite FAM mixtures in Figure 4.9c, show similar δ values with production temperature of the mixtures, across the temperature range. Thus confirming the little effect that the incorporation of zeolites at different production temperatures had on the overall rheological performance of these mixtures.

Furthermore, the effect of foaming technology, with regards to phase angle, can be observed by comparing the isochronal plots of the phase angle for the Mechanical foamed FAM mixtures and the Zeolite FAM mixtures in in Figure 4.9b and in Figure 4.9c, respectively, to that of the Standard FAM mixture at 160ºC, which is also included in these plots. For instance, Figure 4.9b shows that from 15 to 35ºC, there is no significant difference in the phase angles of the Mechanical foamed-FAM mixtures manufactured at 90 and 120ºC and the Standard FAM-160ºC, while the Mechanical foamed-FAM mixture manufactured at 160ºC, exhibits a sharper increase in the δ values at these testing temperatures. Further increase in the testing temperature for the Mechanical foamed-FAM mixtures results in a move towards a more elastic response, as depicted by the lower δ values compared to those of the Standard-FAM mixture manufactured at 160ºC. Similar to what was observed in the δ master curves, at high temperatures (i.e. above 45ºC) the Standard-FAM 160ºC exhibits higher δ values compared to those of the two foaming FAM groups, reflecting an effect on the frequency and temperature on this behaviour in the FAM materials.

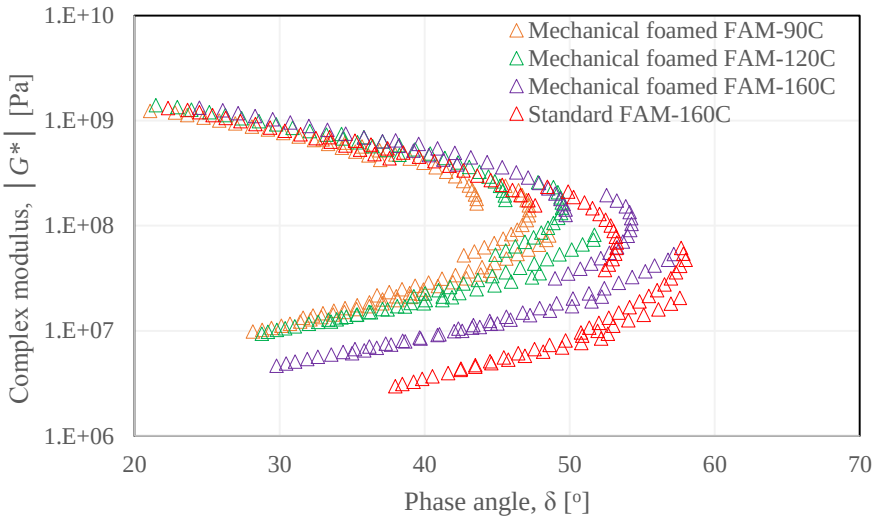
For the Zeolite FAM mixtures, Figure 4.9c shows that this group of mixtures (at all production temperatures) exhibit a higher increase in δ with respect to the Standard-FAM mixture manufactured at 160ºC, from 15 to 45ºC, and they reach the maximum δ value at 45ºC as the Standard-FAM-160ºC mixture, with the same magnitude. Similar to the Mechanical foamed-FAM mixtures at all production temperatures, the effect of further increase in the testing temperature for the Zeolite-FAM mixtures is a move towards a more elastic response, as depicted by the lower δ values compared to those of the Standard-FAM mixture manufactured at 160ºC.

4.2.3 Black Diagrams

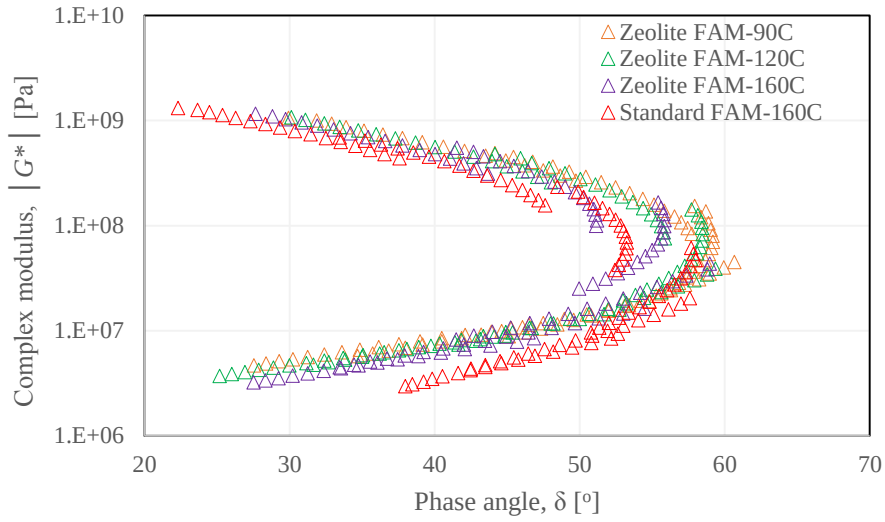
The rheological changes of the three FAM mixtures groups (i.e. Standard, Mechanical foamed and Zeolite mixtures) with production temperature are also described in the black space in Figure 4.10.



a)



b)



c)

Figure 4.10 Black diagrams for: a) Standard-FAM mixtures, b) Mechanical foamed-FAM mixtures, and c) Zeolite-FAM mixtures

As with the master curves, the curves for each foaming FAM group are very distinctive. The curves for the Standard FAM mixtures at all production temperatures are similar, with a slight shift towards lower δ values at 160ºC mixing temperature, meaning that although the changes in the viscoelastic response of these mixtures were not very pronounced, high mixing temperatures of 160ºC increase the elastic behaviour of the resultant mixture. The curves for the Mechanical Foamed-FAM mixtures, show similar δ values at the high $|G^*|$ end, which are related with the upper elastic limit observed in the $|G^*|$ master curves. Near the inflection point however, there is a continuous shift of the curves towards lower δ values with lower production temperatures. This shift of the black diagram is caused by the dual actions of an increase in $|G^*|$ and a decrease in δ, as seen in the corresponding master curves for these mixtures. The black diagrams for the Zeolite-FAM mixtures, on the contrary, are very similar across the $|G^*|$ values, and for all the production temperatures. Thus, confirming as seen from previous results that the production temperature did not present an effect on the rheological behaviour of these mixtures.

Furthermore, looking at the Standard-FAM-160C as the reference control mixture, Figure 4.10b and Figure 4.10c show that in the high $|G^*|$ end, there is no significant difference in the phase angles of the Mechanical foamed and Zeolite-FAM mixtures, and the Standard FAM-160ºC. However after the inflection point, the FAM mixtures with foaming technologies show lower δ values compared to those of the Standard-FAM-160C mixture, which could indicate an impact of the foaming technology on the rheological properties of the materials, as was observed in the previous rheological results.

4.3 Validation of the rheological properties of the FAM mixtures manufactured with different foaming technologies

To validate the behaviour observed in the rheological properties of the FAM mixtures manufactured with the two foaming technologies, a new set of mixtures manufactured with the same FAM mixture design (Section 3.1) and the same foaming production methodologies for the FAM groups (Section 4.1.4) with each foaming technology were produced. In this stage, only a standard-HMA manufactured at 160ºC was produced for comparison purposes. The aggregates type (i.e. limestone) and bitumen penetration grade (i.e. 70/100) remained the same. However, a different bitumen source was utilised compared to the manufacture of the first set of mixtures. According to BS 2000-49:2007 and BS 2000-58:2007 , the penetration grade at 25ºC and softening point of this new bitumen were found to be 89 dmm and 45ºC respectively. It is noteworthy to mention that even if this bitumen comes from a different source, and therefore differences in the chemical properties of each bitumen are expected, both binders are classified within the same penetration grade (i.e. 70/100), which was considered adequate for the purpose of this part of the study.

$|G^*|$ master curves were constructed and are presented in Figure 4.11. These master curves provide the same findings observed in the initial testing plan. For instance, in terms of the $|G^*|$, the master curves for the Mechanical foamed-FAM mixtures in Figure 4.11a, show that low production temperatures of 90 and 120ºC resulted in a clear shift towards higher $|G^*|$ values with respect to the mechanical foamed mixture manufactured at 160ºC, and the Standard-FAM mixture at 160ºC, which is more significant at low frequencies (high testing temperatures). Therefore, these results validate the behavioural trends observed in the initial results of the study, and confirm the hypothesis that this behaviour may be related to the mechanical mixing process used to manufacture these mixtures, which along with the low mixing temperatures (i.e. 90 and 120ºC), generates mixtures with poor binder dispersion and potentially a lower effective binder content, resulting in an increase of the stiffness of these mixtures. Moreover, in terms of the Zeolite-FAM mixtures, Figure 4.11b shows that $|G^*|$ values are very similar for these type of mixtures at all production temperatures, and therefore confirming that the addition of the zeolite did not exhibit significant effects on the rheological properties of these mixtures with production temperature.

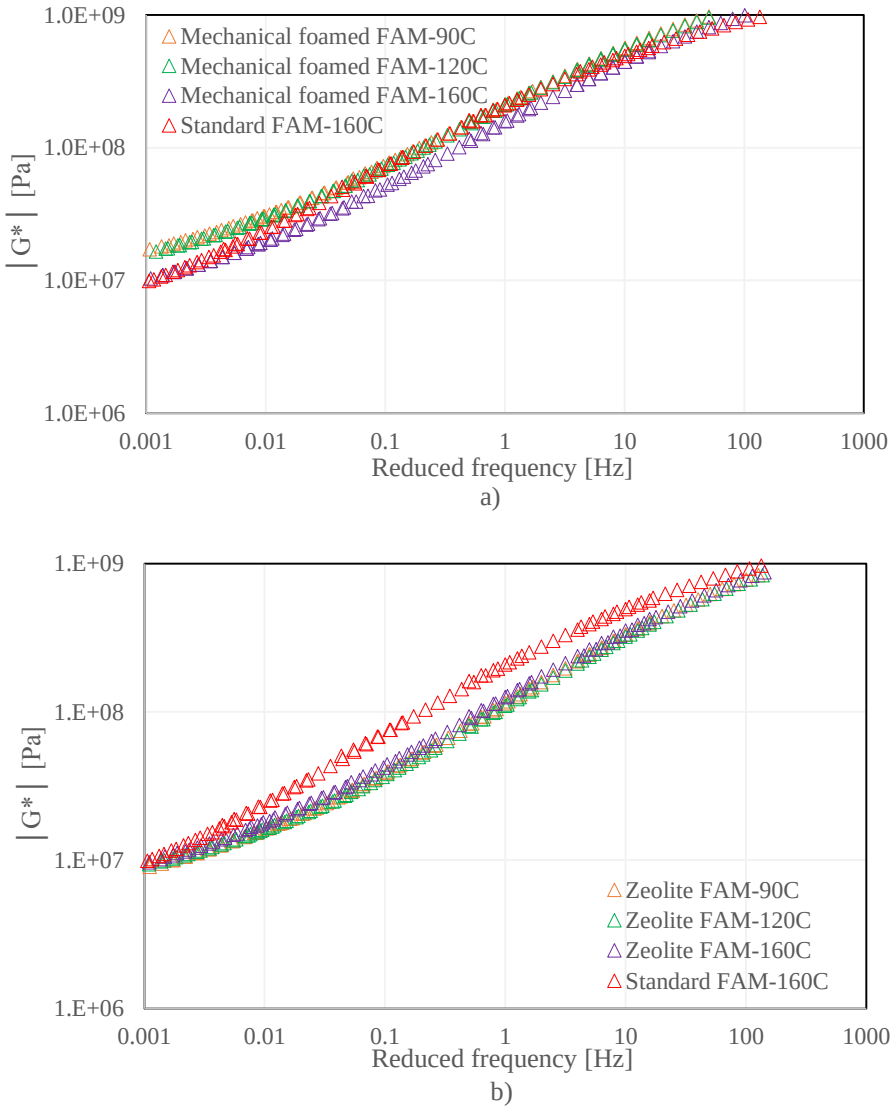


Figure 4.11 $|G^*|$ master curve for: a) Mechanical foamed-FAM mixtures, and b) Zeolite-FAM mixtures, at a reference temperature of 25ºC

Similarly, the phase angle master curves and black diagrams for the new set of mixtures show the same behavioural trends observed in the first set of mixtures (Section 4.2.3), and therefore the results for one foaming FAM group will be presented for conciseness.

The master curves of δ for the Zeolite-RAP mixtures (Figure 4.12) provide the same findings of similar δ values at all production temperatures, and different δ values with reference to the Standard FAM-160C mixture. In addition, the Black diagram curves for the Mechanical foamed-RAP mixtures in Figure 4.13, show a shift towards a more elastic behaviour at lower production temperatures, which is a result of the dual effect of an increase in $|G^*|$ and a decrease in δ at these low production temperatures of 90 and 120ºC, with reference to the Standard FAM-160C and the Mechanical foamed FAM-160C.

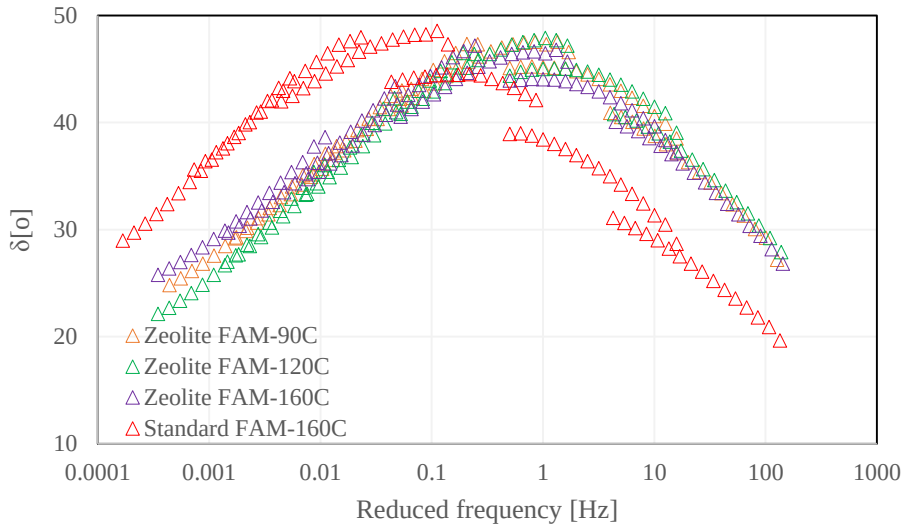


Figure 4.12 δ master curve for Zeolite-FAM mixtures, at a reference temperature of 25ºC

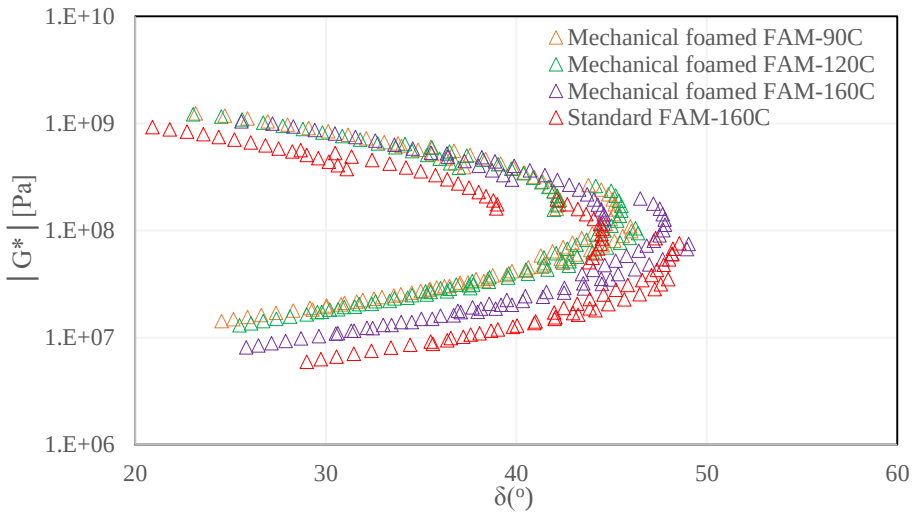


Figure 4.13 Black diagrams for Mechanical foamed-FAM mixtures

These results validate the behavioural trends observed in the old set of mixtures for both foaming FAM groups, meaning that particularly for the Mechanical foamed mixtures, the mixing process plays a significant role in the rheological response of the mixtures. With the aim of further evaluating the effect of binder content and bitumen characteristics on the properties of the Mechanical foamed mixtures, a new set of mixtures with different penetration grade binders and various bitumen contents were produced and the properties of these mixtures were compared to those of the Mechanical foamed-FAM mixtures to get a better understanding of the rheological properties of these materials with production temperature. These results are presented in this section.

4.4 Effect of binder content on the dynamic shear modulus master curves

For this purpose, a new set of mixtures with different penetration grade binders and various bitumen contents were produced using the same aggregate gradation described in Section 3.1. The properties of these mixtures were compared to those of the Mechanical foamed-FAM mixtures manufactured at all the evaluated temperatures to get a better understanding of the rheological properties of these materials with production temperature.

Three additional FAM mixtures were manufactured, and the Standard FAM mixture produced at 160ºC, which is the same mixture presented in the first set of FAM mixtures in Section 4.1.3, was used as a reference mixture for comparison purposes. The three additional mixtures comprise a range of asphalt mixture types in terms of binder grades (i.e. using the same bitumen used to manufacture the Standard FAM mixture, labelled here as "reference bitumen" (RB), using a softer penetration grade bitumen (SB) than the RB bitumen and, a harder penetration grade bitumen (HB) than the RB bitumen), and binder contents of 7 and 10.7%.

All mixtures were manufactured using limestone aggregates and were produced at 160ºC. The characteristics of these mixtures are shown in Table 4-2. This table includes the Standard FAM mixture manufactured at 160ºC, which contains 10.7% binder content. The naming convention for the new set of mixtures is as follows: FAM which denotes the fine aggregate asphalt mixture type, RB, SB and HB denotes the bitumen type in terms of its consistency characteristics, followed by a number that indicates the binder content. For example, FAM-RB-7 corresponds to the FAM mixture, produced with the same bitumen (RB) as the Standard-FAM-160C and containing 7% of bitumen by weight of the total mixture.

Table 4-2 Characteristics of the FAM mixtures with different penetration grade bitumen and binder contents

Mixture	Bitumen Penetration at 25ºC (dmm)	Bitumen content (%)	Aggregates	Air void content (%)
Standard-FAM-160C	70	10.7	Limestone	9.7
FAM-RB-7	70	7.0	Limestone	8.7
FAM-SB-10	145	10.7	Limestone	8.6
FAM-HB-10	38	10.7	Limestone	8.9

The average $|G^*|$ master curves at a reference temperature of 25ºC for these mixtures are presented in Figure 4.14. Each master curve corresponds to the average of two replicate specimens tested for each type of mixture, after observing low variability.

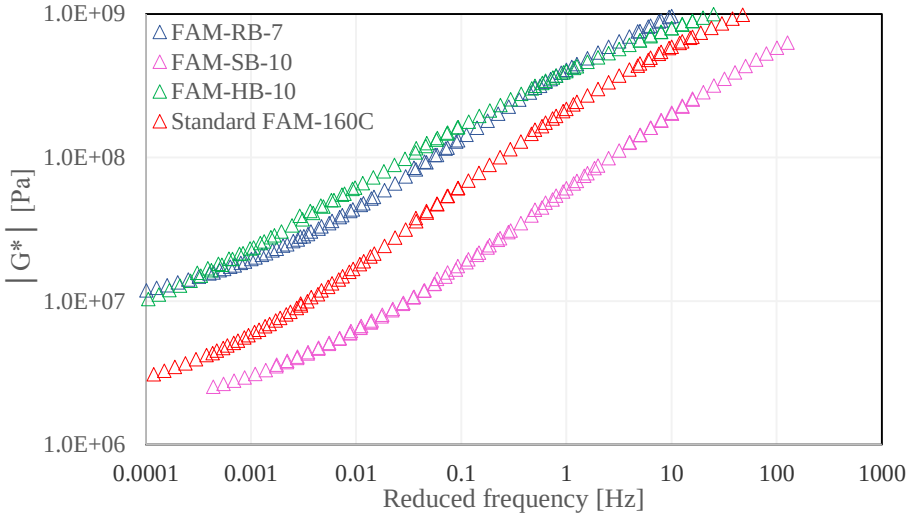


Figure 4.14 $|G^*|$ master curve for FAM mixtures with various binder contents and penetration grade binders

In this figure, the effect of bitumen grade and bitumen content on the $|G^*|$ of the FAM mixtures can be clearly seen. Each FAM mixture shows a different shifting of the $|G^*|$ master curves, compared to the Standard-FAM-160C mixture. For instance, for the same bitumen content used in the Standard-FAM-160C (i.e. 10.7%), the use of a softer bitumen grade resulted in a FAM mixture (FAM-SB-10) with lower $|G^*|$ values, as expected. Similarly, for the same bitumen content (i.e. 10.7%), the FAM mixture manufactured with a harder penetration grade bitumen (FAM-HB-10) exhibited higher $|G^*|$ values as a result of the hardened consistency of the bitumen. Moreover, the FAM-RB-7 mixture, manufactured with the same penetration grade bitumen as the Standard-FAM-160C (i.e. 70/100) but with a lower content (i.e. 7%), exhibited higher $|G^*|$ values to those obtained for the reference Standard-FAM-160C mixture (i.e. which contains 10.7% of the same bitumen). Thus, the stiffness of the FAM mixture increased with lower bitumen content. These results are in good agreement with some results published by other researchers conducted on full asphalt mixtures to evaluate the effect of bitumen and air void content on mix fatigue and stiffness (Harvey and Tsai, 1994).

It is now important to compare the response of the set of FAM mixtures presented in Figure 4.14 to those observed in Figure 4.6b for the Mechanical foamed FAM mixtures, to get a better understanding of the behaviour of the Mechanical foamed FAM mixtures produced at different temperatures. Figure 4.15 combines the $|G^*|$ master curves of the above mentioned mixtures.

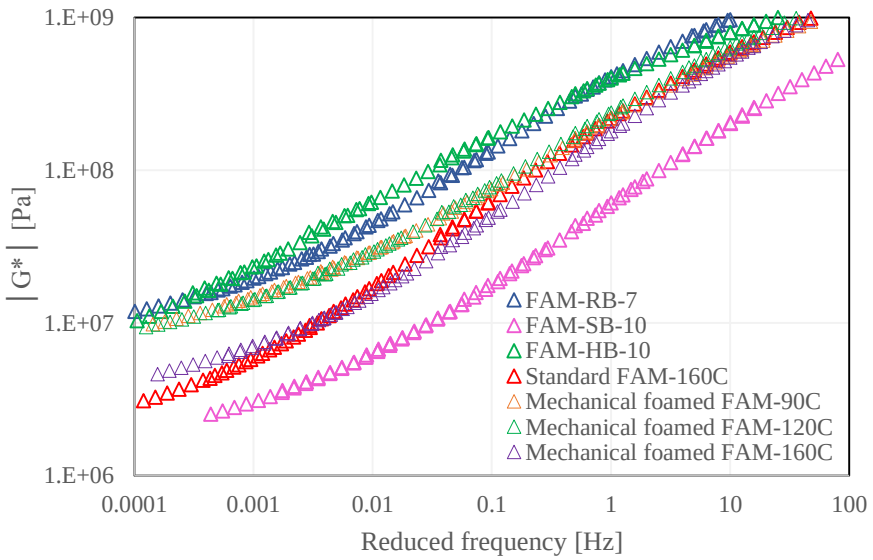


Figure 4.15 Effect of binder content on the G* of the Mechanical foamed-FAM mixtures

From an initial observation of these master curves, it can be seen that at low reduced frequencies, the $|G^*|$ values of the Mechanical foamed mixtures manufactured at 90 and 120°C, approach those of the FAM-RB-7 and FAM-HB-10 FAM mixtures. Figure 4.16, studies this aspect in more detail by presenting the values of $|G^*|$ for all the mixtures at a frequency of 0.001Hz at the reference temperature (25°C).

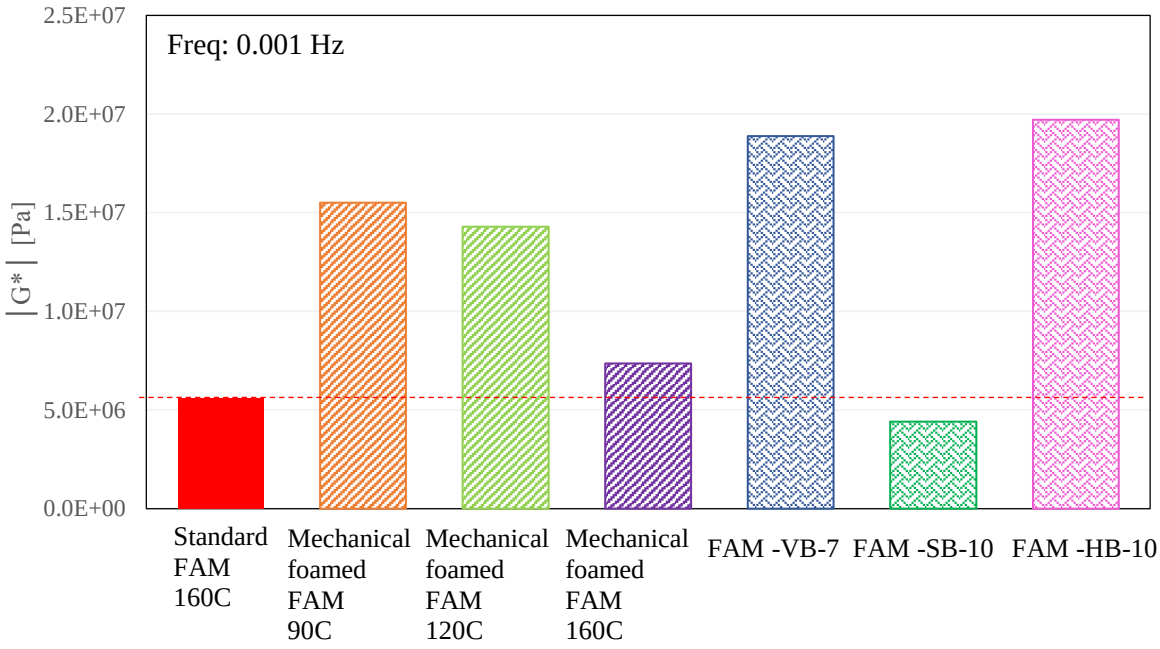


Figure 4.16 Effect of binder content on the $|G^*|$ values of the Mechanical foamed-FAM mixtures

Data in Figure 4.16 corroborates that the $|G^*|$ values of the Mechanical foamed FAM mixtures manufactured at 90 and 120ºC, approach more to those of the FAM-RB-7 and FAM-HB-10 FAM mixtures, than to those of the Standard FAM mixture manufactured at 160ºC, and even less to the FAM-SB-10. For instance the difference between the $|G^*|$ of the Mechanical foamed mixtures manufactured at 90 and 120ºC with the FAM-RB-7 is 19.8 and 27.7% respectively, and with the FAM-HB-10 is 23.8 and 31.8% respectively, while the differences with respect to the Standard HMA are 94.3 and 87.7% for the Mechanical foamed mixtures manufactured at 90 and 120ºC, respectively, and with the FAM-SB-10 it is 111.4 and 105.8% respectively.

These results suggest that the behaviour of the Mechanical foamed FAM mixtures manufactured at 90 and 120ºC, (which were manufactured with a target bitumen content of 10.7%) is similar to that of a FAM mixture manufactured with lower bitumen content (FAM-RB-7), and/or a stiffer bitumen (FAM-HB-10 FAM), which produced a FAM mixture with high $|G^*|$ values. Therefore, it is clear that the mixing process herein used to manufacture the mechanical foamed FAM mixtures, resulted in mixtures with lower effective binder content at low mixing temperatures. Poor bitumen dispersion within the mixtures at these low mixing temperatures (i.e. 90 and 120ºC) and high bitumen loss by means of the mechanical foaming process, were the main factors strongly influencing the final rheological response of these mixtures.

To support this hypothesis, the binder content for the remaining SGC FAM specimens (after coring the DMA FAM testing samples) for both foaming FAM mixtures groups was determined following the BS EN 12697-4:2005, and the results are presented in Table 4-3.

Table 4-3 Bitumen content for both foaming FAM groups of mixtures

Mixture	Design bitumen content (%)	Estimated bitumen content (%)	Difference (%)
Mechanical foamed-FAM-90C	10.7	8.2	(-) 26.5
Mechanical foamed-FAM-120C	10.7	8.4	(-) 24.1
Mechanical foamed-FAM-160C	10.7	9.9	(-) 7.8
Zeolite-FAM-90C	10.7	11.6	(+) 8.1
Zeolite-FAM-120C	10.7	11.6	(+) 8.1
Zeolite-FAM-160C	10.7	-	-

Results in Table 4-3 show that the Mechanical foamed FAM mixtures manufactured at 90 and 120ºC have lower binder contents than the Mechanical foamed FAM mixture manufactured at 160ºC, thus confirming that lower production temperatures resulted in Mechanical foamed FAM mixtures with

less effective binder content. Indeed, the Mechanical foamed FAM mixtures have lower binder contents compared to the Zeolite FAM mixtures as well, which implies that the mixing process is crucial in the rheological performance of the final foaming FAM mixtures and, manual mixing contributes to the even distribution of the bitumen within the mixture, and less bitumen loss.

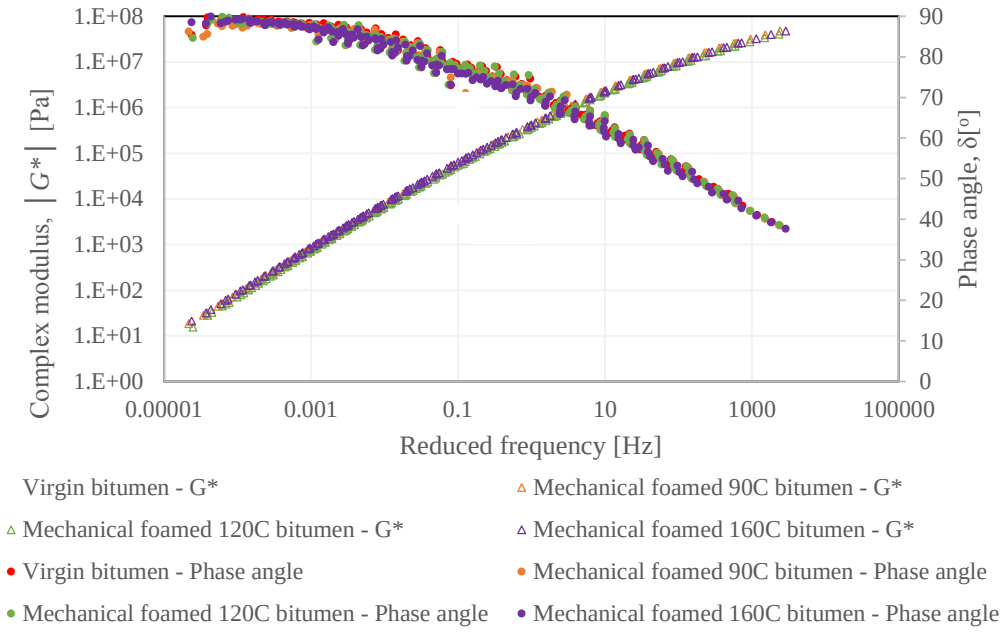
4.5 Rheological characteristics of the binders present in the foamed bitumen mixtures

With the aim of evaluating the differences in the rheological properties of the binders present in the mixtures manufactured with both foaming technologies, the binders of the set of foaming-FAM mixtures prepared to validate the rheological behaviour of the mixtures in Section 4.3 were recovered by distillation following the BS EN 12697-4:2005, and their rheological properties were obtained by means of the Dynamic Shear Rheometer (DSR) analysis.

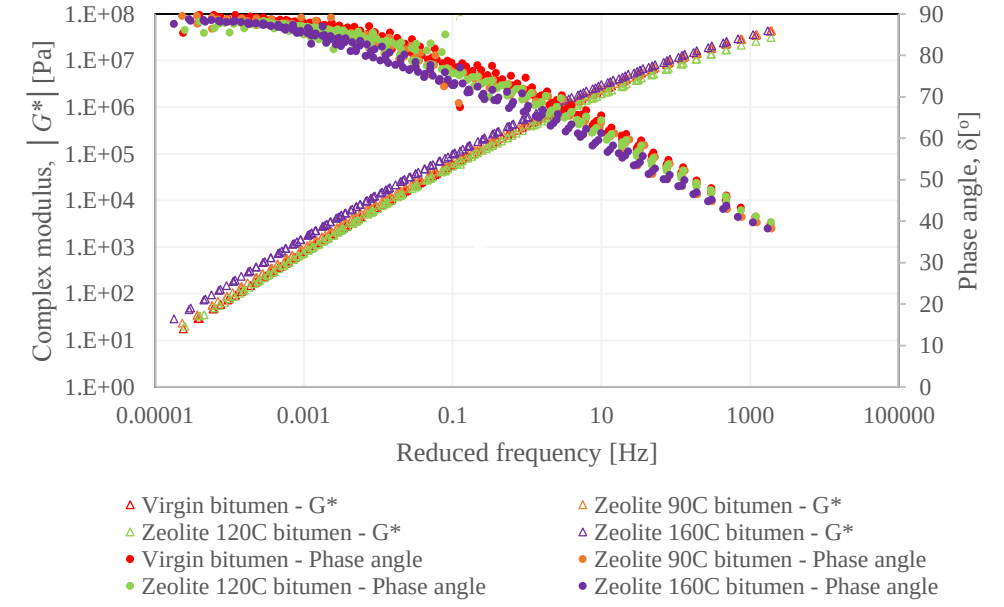
The recovered binders from each FAM mixture were tested in a Bohlin Gemini 200 DSR machine to obtain their $|G^*|$ and δ. Frequency and temperature sweep tests were conducted using the testing conditions described in Section 3.2.2.

4.5.1 Dynamic Shear Modulus Master Curves of the Recovered Bitumens

The master curves in Figure 4.17, reveal the effect of production temperature of the foaming FAM mixtures on the rheological properties (i.e. $|G^*|$ and δ*)* of their corresponding bitumens. For instance, the master curves for the binders recovered from the Mechanical foamed mixtures (Figure 4.17a), show that all the binders presented similar $|G^*|$ and δ values, regardless of the production temperature of the mixtures, and also compared to those of the virgin bitumen, indicating that the rheological properties of these binders did not change during the mixtures production. The master curves for the binders recovered from the Zeolite mixtures (Figure 4.17b), on the contrary, exhibit slight changes in the $|G^*|$ and δ values, with production temperatures of the mixtures. The reason for the slight shifting of the $|G^*|$ and δ master curves with production temperature for the two bitumen groups is hypothesized to be due to potential short-term ageing processes occurring in the binders during the production processes of the mixtures with each foaming technology. For the first group (binders recovered from the Mechanical foamed mixtures), the bitumen is always at 160ºC for foaming purposes, regardless of the final temperature of the mixture. Therefore, no change in the rheological properties are expected in this group of binders. Nevertheless, for the second group (binders recovered from the Zeolite mixtures) the bitumen is heated at the corresponding mixing temperature of the mixtures (i.e. 90, 120 and 160ºC) prior to manufacturing, resulting in different degrees of short-term ageing in the binders.



a)

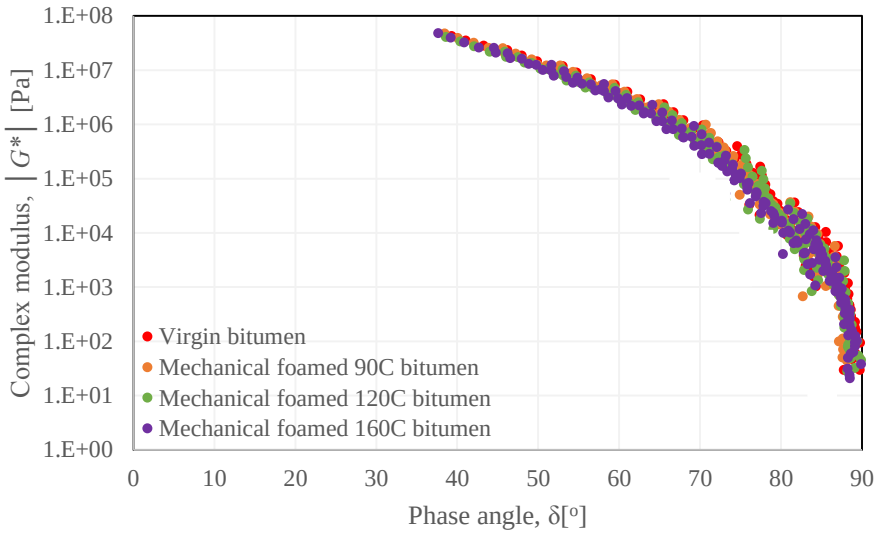


b)

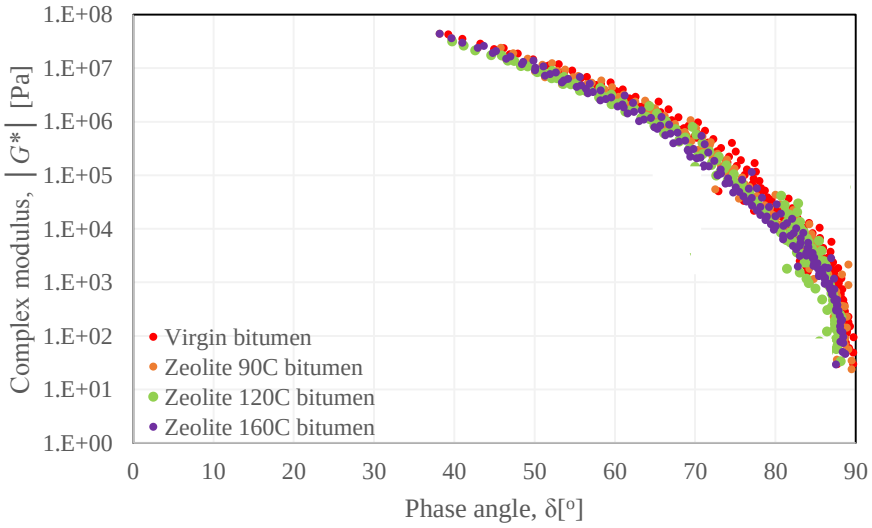
Figure 4.17 |G*| and δ master curve for: a) Recovered binders from Mechanical foamed-FAM mixtures, and b) Recovered binders from Zeolite-FAM mixtures

Furthermore, the black diagram curves for the two groups of recovered binders are presented in Figure 4.18. The curves for the binders recovered from the Mechanical foamed mixtures (Figure 4.18a) did not exhibit important changes in the rheological properties with respect to the production temperature of the mixtures, as was observed in the previous rheological results. The curves for the binders recovered from the Zeolite-FAM mixtures instead (Figure 4.18b), show a slight shift of the

black diagrams towards lower δ values with production temperature, indicating hardening of the bitumen and increase in elastic behaviour due to short term ageing occurring during the production process of these mixtures.



a)



b)

Figure 4.18 Black diagrams for: a) Recovered binders from Mechanical foamed-FAM mixtures, and b) Recovered binders from Zeolite-FAM mixture

4.6 Conclusions

- The foaming action of the zeolite when in contact with the bitumen depends on the properties of the bitumen (i.e. on bitumen viscosity, mainly). This behaviour did not affect the overall performance of the mixtures produced with this technology.

- The rheological response of the Mechanical foamed FAM mixtures was strongly influenced by the mixing process and mixing temperature used to fabricate these mixtures, which generated in this case FAM mixtures with high $|G^*|$ and low δ values (more elastic response) at low production temperatures (e.g. 90 and 120^{o}C), particularly at low frequencies or high temperatures. Poor bitumen dispersion and lower effective binder content were the main reasons associated with this response. These results suggest that using a mixing process that optimises these factors is critical for the fabrication of these mixtures. Furthermore, the practical considerations of altering the mixture design during production include unexpected performance or the presence of early distresses of the mixtures in field where a particular response of the material is required to meet the specific climatic and loading conditions of the area of the project, that were taken into account in the design.

- The production of FBMs by means of a mechanical process at 160^{o}C results in a mixture with similar properties to those of a standard FAM mixture manufactured at 160^{o}C, thus using mechanical foaming for hot applications does not offer benefits with respect to the conventional HMA, and the energy related benefits are no longer obtained.

- The Zeolite containing FAM mixtures did not exhibit significant changes with production temperature, over the range of loading frequencies, indicating low temperature susceptibility. Nevertheless, with respect to the Standard FAM mixture manufactured at 160^{o}C a reduction in $|G^*|$values was more noticeable at higher frequencies. The reduction in stiffness at low temperatures provides the Zeolite mixtures the potential to improve the resistance of cracking and fatigue.

- The DMA testing technique was able to capture the rheological properties of the FAM mixtures manufactured with different foaming technologies including different variables such as production temperature and effective binder content present for each mixture.

- With regards to the bitumen present in the two foaming-FAM mixtures, the different requirements for the materials preparation for the corresponding foaming technology had minor influence on the rheological properties of the binders recovered from the Mechanical foamed mixtures and a slight short-term ageing in the binders recovered from the Zeolite-FAM mixtures. This short-term ageing was not observed to have major influence on the rheological properties of the mixtures where other effects were observed to have a predominant effect on the final mixtures response (e.g. mixing process). However, these results highlight one more time that there are differences in the behaviour of the materials with the foaming technology.

Chapter 5 : Rheological properties of FBMs with RAP material

In Chapter 4, the rheological changes in foamed bitumen mixtures manufactured by means of a mechanical process and by the addition of zeolites into the bitumen were evaluated for half-warm, warm and hot applications. For the former, mixing process and production temperatures were found to be critical in the final properties of the mixtures. For the latter, the zeolites did not generate significant changes in the rheological properties of the final mixtures over the different production temperatures evaluated.

In this chapter, 50% of the components of these foamed bitumen mixtures were changed to incorporate RAP material, and investigate the effects in the rheological properties of the final mixtures. As in Chapter 4, these mixtures were evaluated for half-warm, warm and hot processes. Results indicate that when 50% of the components of the FBMs are replaced by RAP material, a totally different behaviour in terms of the rheological properties of the mixtures is obtained. This means that the rheological response observed for the FBMs in Chapter 4 does not apply when RAP is incorporated.

5.1 Experimental programme

5.1.1 FBM technologies and RAP material

The same bitumen foaming methods for the production of FBMs in Chapter 4 were used. These methods include: 1) a mechanical process that consists of the direct application of cold water into hot bitumen using a laboratory foaming plant and, 2) a regular foamed WMA technology that includes the direct addition of a foaming additive (i.e. zeolite) into the bitumen prior to mixing with the aggregates.

The RAP material used in this study was produced in the laboratory with the aim of controlling the characteristics of its components (i.e. RAP aggregate gradation, bitumen grade, etc.), while reducing issues related with the variability of their properties that could affect the reliability of the results. The laboratory procedure for FAM-RAP production consisted of subjecting a loose sample of FAM mixture to a controlled artificial long-term ageing protocol in the laboratory. Initially, the FAM mixture was manufactured with the same composition (i.e. bitumen content and aggregate gradation)

as the virgin HMA presented in Section 4.1.3, at the same mixing temperature (i.e. 160ºC). Then, the FAM loose mixture was subjected to an ageing protocol which consisted of exposing the FAM loose mixture to heat in the oven for 32 hours (non-continuous ageing: 8h periods over 4 days) at 105ºC, as suggested by Oke (2010). Once the RAP material was produced, the bitumen was recovered by distillation following the BS EN 12697-4:2005. In order to characterise this bitumen, penetration at 25°C (BS 2000-49:2007), softening point (BS 2000-58:2007), rotational viscosity at 150°C (BS 2000-505:2010) and Fraass breaking point (BS 2000-80:2007) tests were performed. The same tests were performed on the virgin bitumen, which is the same as described in Section 4.1 – Chapter 4 used to manufacture the Virgin HMA. The results of both binders are presented in Table 5-1.

Table 5-1 Properties of the 70/100 pen virgin bitumen and recovered bitumen from RAP

Bitumen	Penetration at 25ºC (dmm)	Softening point (ºC)	Rotational viscosity @150ºC (mPa s)	Fraass breaking point (ºC)
Virgin bitumen	73	46	140	-12
Recovered bitumen	38	53	217	-5

5.1.2 Characteristics of the FAM mixtures

Six FAM mixtures manufactured with the two foaming technologies described previously in combination with RAP material were manufactured in the laboratory using three different production temperatures (i.e. 90, 120 and 160ºC). These mixtures include three FBMs produced through a laboratory foaming plant plus RAP material (Mechanical Foamed-RAP), and three FBMs containing zeolites plus RAP material (Zeolite-RAP). For the Mechanical Foamed-RAP mixtures, the bitumen was heated at 160ºC in all cases, and the aggregates were heated 20ºC above the mixing temperature in order to achieve the targeted mixing temperatures (i.e. 90, 120 160ºC). For the Zeolite-RAP mixtures, both bitumen and aggregates were heated to 90, 120 and 160ºC to achieve these target final temperatures of the mixtures.

Furthermore, three control mixtures were manufactured for comparison purposes, which include: a FAM mixture containing 50% RAP material, with no foaming technology labelled as ‘HMA-RAP’, a 100% RAP mixture labelled as ‘100%-RAP’, and a virgin FAM mixture - no RAP material- with no foaming technology, labelled as ‘Virgin-HMA’, which corresponds also to the materials used to produce the RAP material. The three control mixtures were manufactured at 160ºC, and both RAP and virgin materials were manufactured using the same FAM mixture design (i.e. gradation and bitumen content) described in Section 3.1.

Based on the information presented previously, Table 5-2 summarizes the characteristics of the FAM mixtures evaluated. The amount of RAP material required for the 50%RAP mixtures was heated up to the corresponding temperature for 6 hours prior its incorporation with the virgin material. Also, in order to account for the stiffened RAP bitumen present in the mixtures containing RAP, a soft virgin bitumen was used in these mixtures, whose properties were determined using a bitumen blending design as explained in the next section.

Table 5-2 Characteristics of the FAM mixtures with RAP material

Mixture Type	T of the Materials (ºC)			Mixing T (ºC)	Foaming technology
	Bitumen T (ºC)	Aggregates T (ºC)	RAP T (ºC)		
Virgin HMA	160	160	-	160	None
HMA-RAP	160	160	160	160	
100% RAP	160	-	160	160	
Mechanical foamed-RAP-90C	160	110	110	90	Mechanical foaming
Mechanical foamed-RAP-120C	160	140	140	120	
Mechanical foamed-RAP-160C	160	180	160	160	
Zeolite-RAP-90C	90	90	90	90	Zeolite
Zeolite-RAP-120C	120	120	120	120	
Zeolite-RAP-160C	160	160	160	160	

Cylindrical FAM specimens of 50 mm in height by 12.5 mm in diameter were prepared for each FAM mixture following the methodology described in Section 3.2. Frequency and temperature sweep tests were conducted on the cylindrical FAM samples for all the mixtures by means of DMA testing to determine the linear viscoelastic material properties using the testing conditions described in Section 3.2.1. Two specimens were tested after observing low variability in their rheological properties (i.e. differences between $|G^*|$ values below 10%). Since the SGC process results in different air void distribution along the sample, the resultant cylindrical samples contain different air void contents as well. Therefore, the specimens to be tested were carefully selected to have similar air void contents to be able to compare the impact of the components of the mixtures in their mechanical properties. Table 5-3 presents the volumetric characteristics of the tested specimens for each mixture including the measured air void content determined by volumetric calculations.

Table 5-3 Volumetric properties of the tested specimens

Mixture	Section of the sample	Weight (g)	Φ (mm)	h (mm)	Vol (cm^3)	Gmb (g/cm^3)	AV (%)	Average AV (%)
Virgin HMA	A3	12.1	12.5	49.7	6.093	1.986	9.7	9.7
	A5	12.4	12.4	49.7	6.012	2.063	9.7	
100%RAP	A2	12.86	12.4	50.4	6.190	2.089	8.5	8.4
	A4	12.63	12.5	50.6	6.205	2.094	8.3	
HMA-RAP	A3	12.79	12.4	50.6	6.111	2.093	8.4	8.4
	A12	13.00	12.4	51.3	6.195	2.098	8.1	
Mechanical foamed-RAP-90C	A3	12.94	12.5	50.2	6.160	2.100	8.0	8.4
	A12	12.99	12.5	50.6	6.210	2.092	8.4	
Mechanical foamed-RAP-120C	A2	12.74	12.4	50.4	6.086	2.093	8.4	8.3
	A4	12.65	12.4	50.2	6.062	2.087	8.6	
Mechanical foamed-RAP-160C	A1	12.86	12.4	50.8	6.135	2.096	8.2	8.1
	A3	12.98	12.5	50.5	6.197	2.094	8.3	
Zeolite-RAP-90C	A3	13.30	12.5	51.6	6.332	2.100	8.0	8.2
	A12	13.10	12.5	51.2	6.283	2.085	8.7	
Zeolite-RAP-120C	A12	13.14	12.6	50.5	6.297	2.087	8.6	8.1
	A16	12.74	12.4	50.4	6.086	2.093	8.4	
Zeolite-RAP-160C	A13	12.91	12.5	50.1	6.148	2.100	8.1	8.2
	A17	12.84	12.4	50.8	6.135	2.093	8.4	

5.1.3 Bitumen blend design

For the mixtures containing 50% RAP, equations (1) and (2) were used to determine the properties of the expected final bitumen blend to predict the required properties of the virgin bitumen to obtain a mixture of 70/100 pen (i.e. equal to the virgin bitumen in the HMA mixture for comparison). Table 5-4 shows the properties of each component of the final bitumen blend.

Table 5-4 Blend design for the RAP-FBMs

Bitumen	Penetration at 25°C (dmm)	Softening point (°C)
Estimated virgin bitumen	141	39
Recovered bitumen from RAP	38	53
Final expected bitumen blend	73	46

Following these results, a virgin bitumen of 145 dmm penetration at 25ºC, softening point of 41ºC, and Fraass breaking point of -13ºC, was used for the soft virgin bitumen in the HMA-50% RAP mixture and as the foamed bitumen present in the RAP- FBM materials. Based on the previous calculations, it is expected that this virgin bitumen when blended with the RAP bitumen, will produce a final binder with an equivalent 70/100 penetration. Therefore, for the production of these mixtures, 50% of the amount of virgin materials (i.e. virgin bitumen and virgin aggregates) were replaced by RAP material, targeting the same gradation and bitumen content described in Section 3.1.

5.1.4 FAM mixing

For the mechanical foamed-RAP mixtures, mixing was carried out using a Hobart type mixer with a flat type agitator as explained in Section 4.1.4. First, the amount of RAP material and virgin aggregates, previously heated at the correspondent temperature as shown in Table 5-2, were added to the Hobart mixer bowl and mixed for approximately 30 seconds. Then, while the aggregate mixing was still in progress, foamed bitumen was sprayed out from the nozzle into the bowl and directly mixed with the pre-heated aggregates for up to 3 minutes until all the aggregates were visually fully coated. The bitumen foaming temperature was 160ºC in all cases, and the same foaming water content used for the manufacture of the mechanical foamed mixtures in Section 4.1 was used (i.e. 3% by mass of bitumen). The mixtures were then placed in the SGC mould for further compaction, without reheating the material.

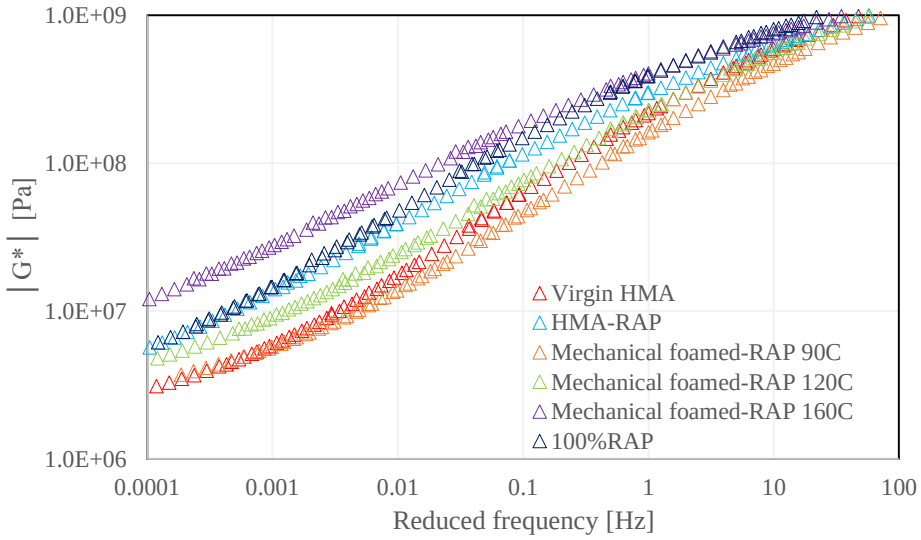
For the preparation of the Zeolite-RAP mixtures, hand mixing was carried out in a heated mixer, which was heated at the corresponding mixing temperature of the mixture. First, as in the Mechanical foamed-RAP mixtures, the amount of RAP material and virgin aggregates, previously heated at the correspondent temperature as shown in Table 5-2, were added to the mixer and mixed manually for approximately 30 seconds. Then the same process as explained in Section 4.5.2 was followed. In summary, the process consisted of pre-blending Advera® (at room temperature) with the corresponding pre-heated bitumen at 90, 120 and 160ºC, for further incorporation to the RAP-virgin aggregates. Then, hand mixing was performed until the aggregates were fully coated. The mixtures were then placed in the SGC mould for further compaction, without reheating the material. The dosage rate of Advera® was the same used for the preparation of the Zeolite mixtures in Section 4.5.2, which corresponds to 5% by weight of the bitumen.

For the preparation of the Virgin-HMA and the HMA-50%RAP mixtures, hand mixing was performed as well until the fine aggregates were fully coated. These mixtures were placed in the oven at 140ºC for 1 hour before compaction following the BS EN 12697-35:2016.

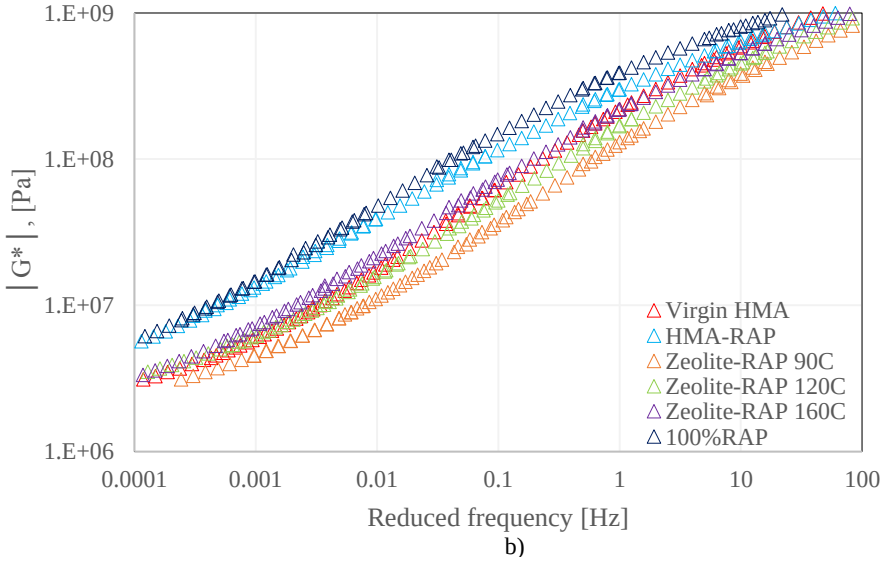
5.2 Dynamic Mechanical Analysis

5.2.1 Complex Modulus Master Curves

The $|G^*|$ master curves at a reference temperature of 25ºC for the two foaming-RAP groups of mixtures are presented in Figure 5.1. In these curves, the Virgin HMA mixture - no RAP material, no foaming technology –, the HMA-RAP mixture– no foaming technology –, and the 100%RAP mixture are included for comparison as reference mixtures, and also to evaluate blending between the RAP and virgin binders.



a)



b)

Figure 5.1 $|G^*|$ master curve for: a) Mechanical foamed-FAM mixtures, and b) Zeolite-FAM mixtures

These figures reveal the effect of production temperature on $|G^*|$. In both foaming-RAP FAM groups, the mixtures exhibited a better mechanical capacity as the production temperature increased, as depicted by a shift towards higher $|G^*|$ values with production temperature, over the range of reduced frequencies. However, each mixture exhibits a different shifting of $|G^*|$ towards higher or lower values with respect to the Virgin HMA, with production temperature, and also over the range of reduced frequencies.

The different shifting of the $|G^*|$ with reference to the Virgin HMA, could be partially explained by the actual level of blending occurring between the hard bitumen from the RAP and the soft virgin bitumen, which can also be reflected in Figure 5.1. In this figure, the behaviour (i.e. $|G^*|$ values) of the virgin HMA and the RAP material (i.e. 100% RAP mixture) are exhibited, and are significantly different. If complete blending would have occurred between the virgin bitumen and the bitumen from RAP in the HMA-RAP mixture (manufactured at 160ºC), the resultant mixture should present similar properties to that of the Virgin-HMA since, in theory, the combination of the soft virgin bitumen used for the preparation of these materials, and the bitumen from RAP would produce a mixture with similar properties to that of the Virgin HMA. However, the $|G^*|$ values of the HMA-RAP mixture are higher, ending up with a mixture with similar properties to that of the 100% RAP mixture, suggesting that complete blending between the old and new binders did not occur. This means that the behaviour of this mixture is primarily dominated by the hardened bitumen from RAP, and little interaction with the virgin bitumen was obtained. The Mechanical foamed-RAP mixture manufactured at 160ºC exhibits also high $|G^*|$ values, even higher than those reported for the HMA-RAP mixture (Figure 5.1a). This behaviour breaks the analogy of complete blending since the properties of the Mechanical foamed-RAP mixture at 160ºC is out of the range of the properties of its constitutive components. However, these results imply that as observed in the HMA-RAP mixture, full blending was also not obtained in this mixture, and indicates that at this elevated temperature of 160ºC a different mechanism is occurring within the FAM mixtures.

One hypothesis that may explain this behaviour is the internal structure of the FAM-RAP materials. Unlike conventional full asphalt mixtures, in which aggregates of different sizes coexist (i.e. coarse and fine particles) in these FAM materials, only fine particles (i.e. below 1 mm) are present. Therefore, in full asphalt mixtures when RAP is incorporated and it behaves as a “black rock”, where incomplete blending occurs, it acts as an intrusion of solid particles that influences the mix volumetrics, properties, and performance through its aggregate gradation properties (McDaniel and Anderson, 2001); However, within FAM materials, when incomplete blending occurs, it is speculated that the FAM-RAP particles (having similar size) bond together with the bitumen forming clusters that influence the behaviour of the final mixture through their hardened bitumen properties, which

could explain the increased stiffness observed in the Mechanical foamed-RAP manufactured at 160ºC and the HMA-RAP mixtures.

The Zeolite-RAP mixtures manufactured at 90, 120 and 160ºC and the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC, exhibited a softening effect with respect to the HMA-RAP, as depicted by lower |G*| values. Furthermore, although these mixtures exhibited a different shift of $|G^*|$ values with respect to the Virgin-HMA, these mixtures could still be comparable to the Virgin HMA. These results suggest that the level of blending between old-new materials occurred to a different extent in each mixture, depending on the final mixing temperature and production process (i.e. foaming technology).

Following these results, it is theorised that three different component systems are present in these FAM mixtures consisting of: 1) the soft virgin bitumen, 2) part of the hard bitumen from RAP and, 3) the actual bitumen blend composed of the previous two, where the amount of the two last components depends on the mixing temperature of the mixtures, and the foaming technology (i.e. particularly mixing process). Taking into account that all the mixtures contain the same amount of virgin and RAP materials, with the same target bitumen content, and that the level of blending depends on the mixing temperature and mixing mechanism as described previously, these results also imply that the effective binder content in each mixture manufactured at the evaluated temperatures is also different. This observation is important since it suggest that the properties of the final mixtures are affected not only by the rheological properties of the bitumen blend but by their effective binder content, which could also explain the different shifting of the master curves. Figure 5.2. shows a schematic representation to illustrate this concept.

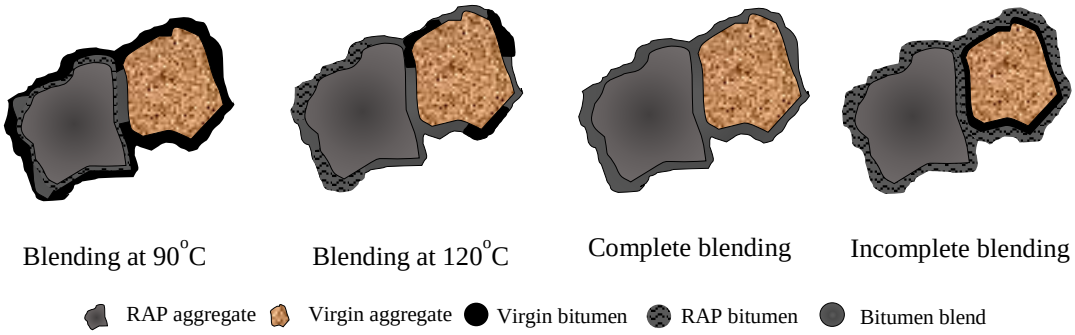


Figure 5.2 Schematic representation of blending between RAP-virgin binders with temperature

The final properties of the two foaming FAM groups in combination with RAP material with production temperature are hypothesised to be influenced by the mixing process and the preparation of the materials for each foaming technology, as mentioned previously. It is speculated that manual mixing contributes to a better distribution of the RAP-virgin bitumen blend within the mixture

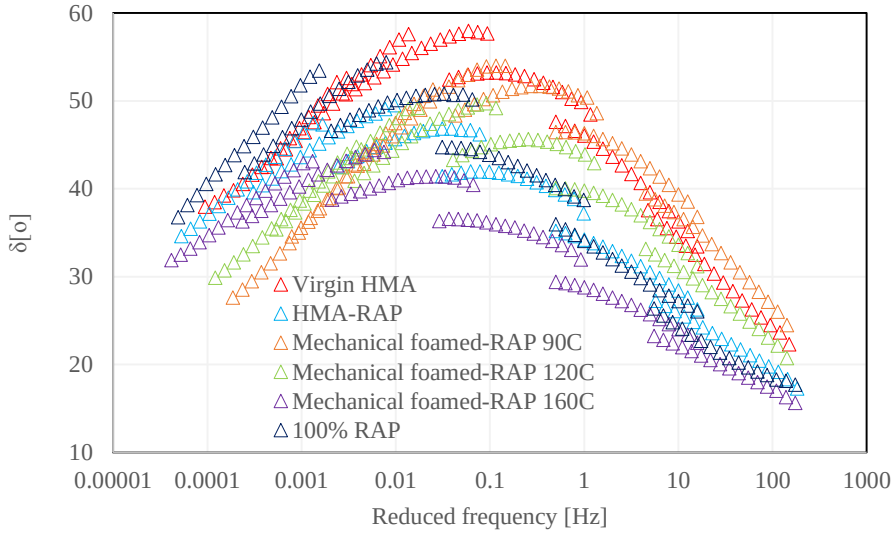
compared to mechanical mixing. In fact, the Zeolite-RAP mixtures exhibited closer $|G^*|$ values to those of the Virgin-HMA, than the Mechanical foamed-RAP mixtures, where mechanical mixing was performed.

5.2.2 Phase Angle Master Curves

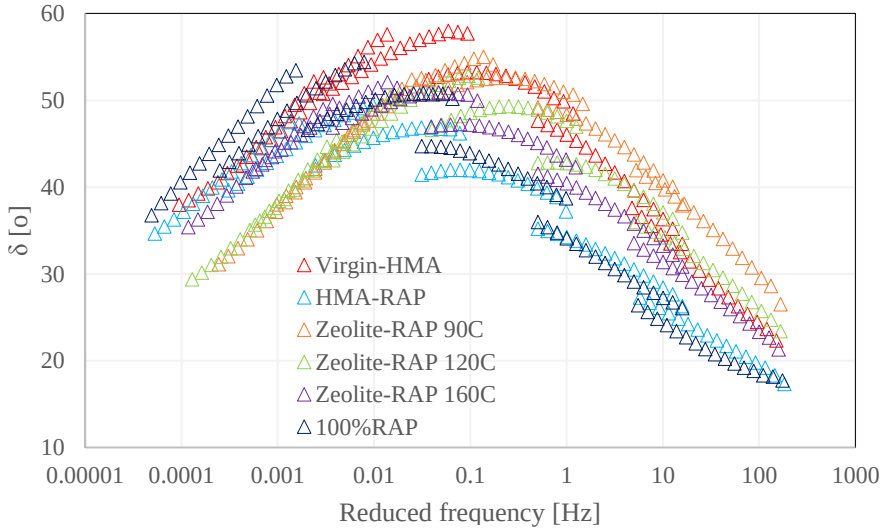
Figure 5.3 shows the δ master curves for the two groups of mixtures. These curves have been produced by using the same shift factors as the $|G^*|$ master curves. As in the $|G^*|$ master curves, these curves include the Virgin HMA mixture, the HMA-RAP mixture, and the 100%RAP material for comparison purposes.

The δ master curves for the two foaming-RAP mixture groups show a clear decrease in δ values with production temperature (more elastic response), at high reduced frequencies (or low testing temperatures), thus indicating that production temperature affects the viscous component of the bitumen present in the mixtures, as could be expected. For the same amount of RAP material, the production temperature in the two foaming-RAP mixture groups increased $|G^*|$, as observed previously in the master curves, and decreased δ leading to a stiffer and more brittle material at high reduced frequencies (low testing temperatures). Low production temperatures as 90 and 120ºC for the Mechanical-foamed technology and all production temperatures 90-160ºC for the zeolite-based technologies resulted in RAP mixtures with almost similar rheological behaviour (master curves overlapped) with the virgin mixture, meaning a better or similar thermal cracking or fatigue resistance for these mixtures. However at low reduced frequencies (high temperatures), the δ master curve for the two foaming-RAP mixtures groups exhibit an increase in δ values with production temperature, but lower δ values with respect to the Virgin HMA, thus indicating a more viscous material. However, the response of mixtures at these low frequencies could be an effect not only of the loading frequency, but also of the complex behaviour of the materials as a result of the discontinuous curves after the shifting process of the master curves.

The HMA-RAP, the Mechanical foamed-RAP mixture manufactured at 160ºC and the 100%RAP mixtures exhibit an increased elastic response at high reduced frequencies (or low testing temperatures), meaning that producing a 100% RAP mixture and incorporating RAP at 160ºC could negatively affect the fatigue and thermal cracking resistance of the mixtures. Similarly, at lower reduced frequencies, the δ values tend to overlap or present a more viscous behaviour with production temperature, which could be in part a result of the shifting of the isothermal data.



a)



b)

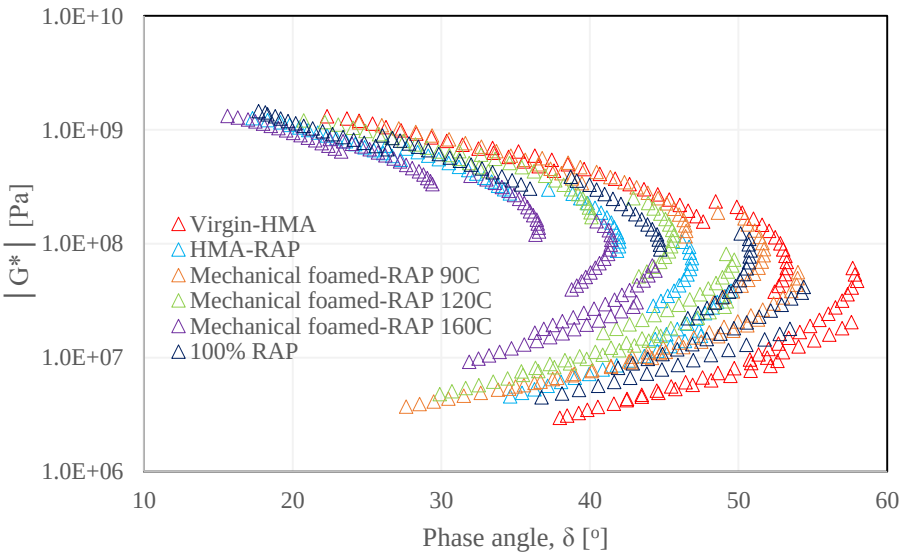
Figure 5.3 δ master curve for: a) Mechanical foamed-FAM mixtures, and b) Zeolite-FAM mixtures, at a reference temperature of 25ºC

5.2.3 Black Diagrams

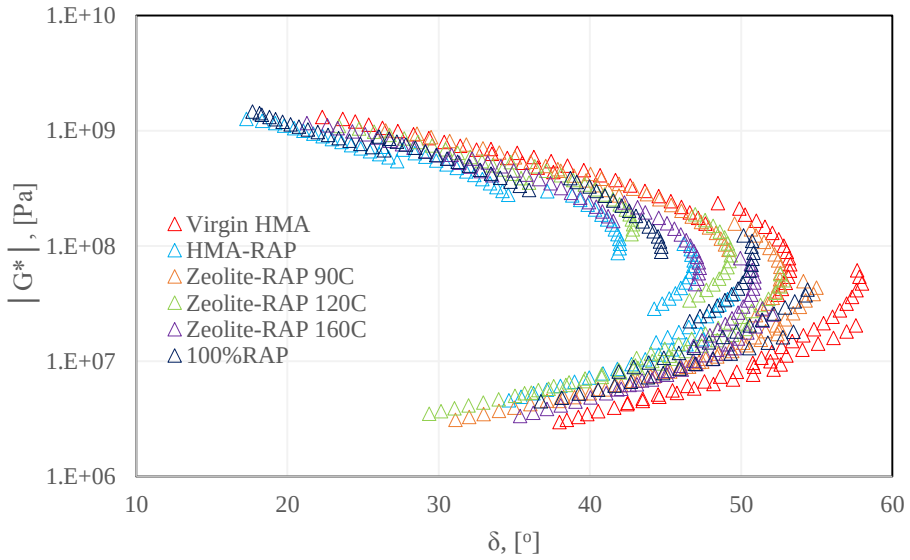
The complete rheological behaviour for the two FAM groups is presented in the black space in Figure 5.4. In black diagrams, the data that is in the high-end of the plot is at low temperatures and the data that is at the lower end of the plot is at high temperatures. At the high-end of the plot, the curves for the Mechanical foamed-RAP mixtures in Figure 5.4a, show lower δ values with production temperature, and also with reference to the Virgin HMA. This reduction in the δ values at similar $|G^*|$ values indicate an increase in the elastic response of the materials, and consequently an increase in their brittle behaviour. After the inflection point, the shift towards a more elastic response becomes more evident. This shift of the black diagram curves is a result of the dual effect of increase

in $|G^*|$ and decrease in δ with production temperature, as was also observed in the corresponding master curves. The Zeolite-RAP mixtures, on the other hand, exhibit similar δ values across the $|G^*|$ values for all with less change in the viscoelastic properties with production temperature.

Furthermore, the two foaming FAM groups with RAP material, exhibit similar phase angle values to those of the Virgin HMA mixture at high $|G^*|$ values. The similarity of results at high $|G^*|$ values relates to the upper limiting elastic stiffness of all the FAM mixtures which will tend to be the same (or at least similar). After the inflection point, the foaming FAM groups with RAP material show lower δ values compared to those of the Virgin-HMA, indicating the impact of the inclusion of RAP in the mixture. The same trend is observed for the HMA-RAP mixture which is similar in composition but without the use of a foaming technology. This behaviour shows that part of the viscous component of the mixtures is decreased by the incorporation of RAP material. Due to the increased elastic response of the bitumen present in the RAP material, the behaviour of mixtures containing RAP will naturally differ from those of the virgin mixture.



a)



b)

Figure 5.4 Black diagram for: a) Mechanical foamed-FAM mixtures, and b) Zeolite-FAM mixtures

5.2.4 Statistical analysis of $|G^*|$ and δ

In order to quantify the significance of the incorporation of RAP material at different production temperatures into FBMs produced with both foaming technologies, the $|G^*|$ and δ raw data at all tested frequencies and temperatures were analysed statistically. The analysis was conducted using independent sample t-tests with a confidence interval of 95%, by comparing the $|G^*|$ and δ raw data obtained from the sweep frequency and temperature tests of the samples tested for each mixture to that of the Virgin HMA. Table 5-5, presents the results at two selected frequencies and at all testing temperatures. Appendix B contains the detailed procedure followed to perform the statistical analysis.

In the analysis the null hypothesis is that the means of each RAP mixture with reference to the Virgin HMA are not significantly different (e.g. $|G^*|$ of Virgin HMA and HMA-RAP are not significantly different). A *p*-value equal to or less than 0.05 are statistical significant (green shaded values in Table 5-5). Otherwise, results yielding a *p*-value above 0.05 are considered not statistical significant.

Table 5-5 Statistical Analysis of Dynamic Complex Modulus at 3.162 and 10Hz

Mixtures comparison	T[°C]	*p-value*				Group
		|*G**|		δ		
		3.162 Hz	10 Hz	3.162 Hz	10 Hz	
Virgin HMA to 100%RAP	15	0.007	0.009	0.010	0.036	A
	25	0.007	0.007	0.014	0.027	
	35	0.005	0.005	0.039	0.036	
	45	0.005	0.003	0.155	0.086	
	55	0.007	0.003	0.593	0.339	
	65	0.011	0.004	0.627	0.704	
Virgin HMA to HMA-RAP	15	0.450	0.311	0.016	0.052	A
	25	0.118	0.308	0.018	0.037	
	35	0.022	0.036	0.023	0.026	
	45	0.005	0.004	0.031	0.021	
	55	0.004	0.003	0.043	0.024	
	65	0.014	0.006	0.231	0.074	
Virgin HMA to Mechanical foamed RAP 160°C	15	0.060	0.657	0.001	0.005	A
	25	0.003	0.017	0.001	0.002	
	35	0.000	0.001	0.001	0.001	
	45	0.000	0.000	0.001	0.001	
	55	0.000	0.000	0.001	0.001	
	65	0.000	0.000	0.010	0.002	
Virgin HMA to Mechanical foamed RAP 120°C	15	0.186	0.058	0.364	0.510	B
	25	0.781	0.395	0.205	0.367	
	35	0.293	0.449	0.096	0.137	
	45	0.052	0.051	0.052	0.057	
	55	0.018	0.014	0.016	0.022	
	65	0.018	0.012	0.025	0.014	
Virgin HMA to Mechanical foamed RAP 90°C	15	0.035	0.040	0.133	0.237	B
	25	0.068	0.046	0.445	0.298	
	35	0.198	0.116	0.759	0.914	
	45	0.844	0.582	0.201	0.288	
	55	0.450	0.584	0.036	0.059	
	65	0.280	0.297	0.022	0.025	
Virgin HMA to Zeolite RAP 90°C	15	0.002	0.001	0.370	0.581	B
	25	0.014	0.005	0.168	0.300	
	35	0.069	0.031	0.117	0.105	
	45	0.401	0.195	0.146	0.078	
	55	0.984	0.770	0.255	0.100	
	65	0.685	0.675	0.499	0.213	

Mixtures comparison	T[°C]	*p-value*				Group
		|G*|		δ		
		3.162 Hz	10 Hz	3.162 Hz	10 Hz	
Virgin HMA to Zeolite RAP 120°C	15	0.013	0.004	0.566	0.551	B
	25	0.060	0.021	0.791	0.771	
	35	0.278	0.132	0.333	0.540	
	45	0.990	0.675	0.119	0.154	
	55	0.459	0.640	0.022	0.037	
	65	0.345	0.378	0.021	0.015	
Virgin HMA to Zeolite RAP 160°C	15	0.081	0.017	0.054	0.069	B
	25	0.544	0.177	0.286	0.180	
	35	0.456	0.694	0.954	0.844	
	45	0.085	0.069	0.350	0.396	
	55	0.059	0.032	0.143	0.128	
	65	0.152	0.061	0.099	0.066	

From the statistical analysis results, the mixtures were grouped together according to their statistical significance with reference to the Virgin HMA. For instance, mixtures that fall into group A, show statistical significance with reference to the Virgin HMA, and those that are not statistically different from the Virgin HMA, fall into group B. For example, the 100%RAP mixture, HMA-RAP mixture and the Mechanical foamed-RAP mixture manufactured at 160°C fall into group A. These mixtures show *p*-values less than 0.05 at most of the temperatures and loading frequencies. At low testing temperatures, particularly at 15°C (corresponding to high reduced frequencies in the master curves), the HMA-RAP and Mechanical foamed-RAP mixture at 160°C show similarity with the Virgin HMA, which could be related to the maximum stiffness of the mixture, which tends to be similar within the mixtures. The Mechanical foamed-RAP mixtures manufactured at 90 and 120°C, fall into group B as the *p*-values show that there is no statistical difference with reference to the Virgin HMA at most of the testing temperatures and frequencies. At low temperatures (15-25°C) the Mechanical foamed-RAP manufactured at 90°C show statistical significance from the Virgin HMA. The Mechanical foamed-RAP mixture manufactured at 120°C show statistical significance from the Virgin HMA at 55 and 65°C (corresponding to low reduced frequencies in the master curves). The heterogeneity of the mixtures must be contributing to these differences. The Zeolite-RAP mixtures at all production temperatures fall into group B. It is interesting to notice from the statistical results for these mixtures, that although there are some *p*-values that are higher than 0.05 (particularly at low temperatures 15-25°C), meaning that there are still some differences with respect to the Virgin HMA, there is a tendency of a decrease in significance differences with production temperature (lower values of *p* below 0.05), which could imply that higher production temperatures for the mixtures manufactured by

the addition of zeolites (i.e. hand mixing), produce more homogeneous mixtures, thus reducing the variability of the mix.

The statistical analysis results corroborate the initial observations made from the $|G^*|$ master curves in Section 5.2.1. For instance, the statistical significance of the HMA-RAP and the Mechanical foamed-RAP 160ºC mixes, with respect to the Virgin HMA means that the properties of the former mixtures are not similar to those of the Virgin HMA, thus confirming that complete blending between the old-new binders did not occur in these mixtures, as was explained previously. On the other hand, the little statistical significance shown for the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC, and the Zeolite-RAP mixtures produced at all temperatures (i.e. 90-160ºC), with respect to the Virgin HMA, means that the properties of these mixtures are comparable to those of the Virgin HMA. Also, as was discussed previously, these results confirm that the production temperature, and foaming process (i.e. particularly mixing) influence the degree of interaction between the old-new binders thus, affecting the rheological response of the final mixtures.

5.3 Rheological Indices

To further evaluate the rheology of the two foaming-RAP mixture groups, two different rheological indexes were calculated from the data provided by the frequency sweeps and accordingly to the methodology provided in the literature review Section 2.5.5. Glover-Rowe parameter and the Crossover frequency ($-\beta/\gamma$) vs. Relaxation spectra (γ). These indexes provide a useful means to track the relative changes in the rheological properties of the RAP-FBMs with production temperature, and also to relate to their performance, in terms of age hardening as explained in Chapter 2. Figure 5.5 shows the *G-R* parameter for the two groups of mixtures, including also the Virgin HMA and the HMA-RAP mixture, as reference mixtures. This parameter was calculated by means of equation 21.

The Glover-Rowe parameter analysis, shown in Figure 5.5, illustrates that initially, the Virgin HMA is at the bottom right of the plot, with low *G-R* parameter and high δ value, as expected. When 50% of the components of this mixture are being replaced by 50% of RAP material (i.e. HMA-RAP mixture), the response of the material moves towards the upper left of the plot, as a result of the stiffening effect provided by the RAP material, producing a more elastic (brittle) material. In terms of the 100%RAP material, the *G-R* parameter analysis shows that this mixture exhibits similar *G-R* values to that of the HMA-RAP mixture, as was also observed in the $|G^*|$ master curves of these materials, and described before. However, the 100%RAP exhibits a higher δ value, even compared to those of the rest of the RAP-FBMs. Taking into account that the G-R parameter is calculated at a

frequency of 0.005rad/sec, which corresponds to the low frequency end in the master curves, the results observed here for the phase angle match those observed in the phase angle master curves (i.e. increase of viscous response as a result of the effect of temperature and frequency), thus affecting the expected tendency of this material.

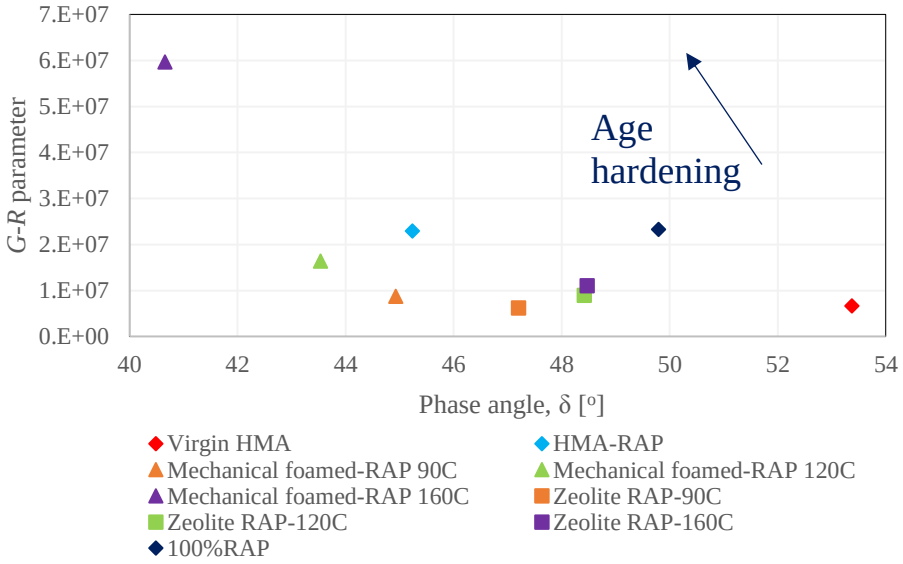


Figure 5.5. Glover-Rowe parameter for virgin and FAM mixtures containing RAP and different foaming technologies at 15ºC and 0.005 rad/sec

Figure 5.5, also shows that the rheological response for the two groups of mixtures show different values due to their different rheological characteristics as seen in the previous results. Indeed, the Mechanical foamed-RAP mixtures undergo more changes in the rheological response with production temperature compared to the Zeolite-RAP mixtures, as the measured *G-R* parameter of these mixtures, have larger increases with production temperature than the Zeolite-RAP mixtures. The Mechanical foamed-RAP at 160ºC shows the highest G-R and lowest δ values, compared to the rest of the mixtures and also to the HMA-RAP mixture (manufactured at the same mixing temperature), thus indicating an increase in the overall hardness and more brittle behaviour of this mixture, which can lead to a poor long-term performance in terms of fatigue and thermal cracking resistance.

Furthermore, to determine the –β and γ values for all the mixtures, data from the $|G^*|$ master curves for the two foaming FAM groups with RAP material, was fitted using the sigmoidal function by means of equation 20. Figure 5.6 shows the cross-plot of the – β/γ and γ values for the two groups of FAM foaming mixtures with RAP material including also the Virgin HMA, the HMA-RAP and the 100% RAP mixtures for comparison purposes.

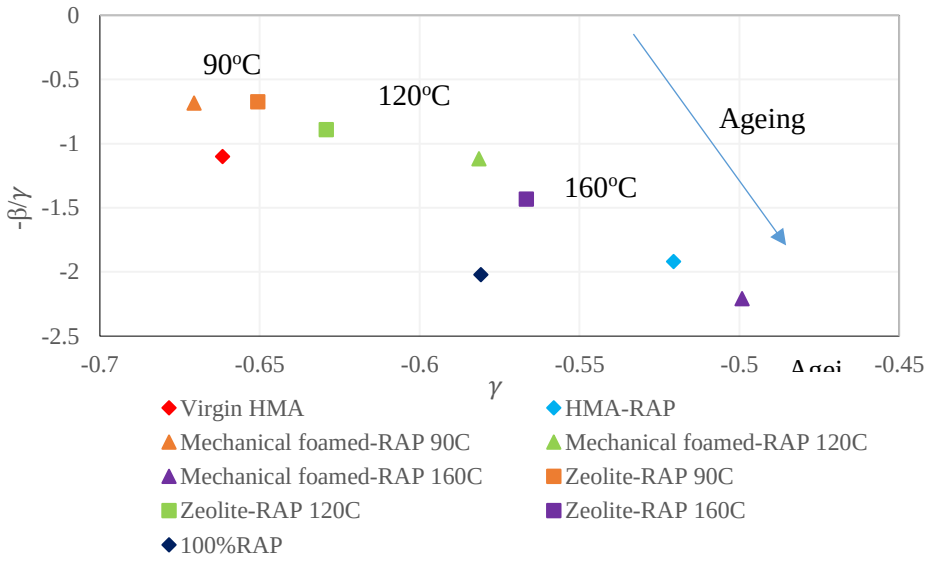


Figure 5.6 Crossover frequency parameter vs. relaxation spectra for virgin and FAM mixtures containing RAP and different foaming technologies

As asphalt materials age, the $|G^*|$ master curve tends to exhibit a characteristic flattening effect (Mensching et al. 2015). In this regard, the γ parameter quantifies the slope of the $|G^*|$ master curve, meaning that higher values of this parameter (lower absolute values) are related to more age hardening in the mixtures due to the flattening effect of the master curve (Mensching et al. 2015). On the other side, β parameter controls the inflection point position of the flattening effect of the master curve after ageing (calculated as $10^{-\beta/\gamma}$), which moves towards lower frequencies, with increase in RAP or ageing of asphalt mixtures (Mensching et al. 2015). Indeed, Figure 5.6 shows that similarly to what has been observed in the Glower-Rowe analysis, both foaming-RAP groups of mixtures exhibit a relative position with mixing temperature. As the mixing temperature increases the mixtures move towards the lower right of the plot (lower values of both parameters), indicating an increase in the hardness of the mixtures. At a mixing temperature of 160ºC, both the Mechanical foamed-RAP mixture, and the HMA-RAP mixture exhibit the highest γ values indicating that these mixtures are expected to be more susceptible to cracking. Furthermore, from this figure the benefit of a low mixing temperature of 90ºC, and the absence of RAP material (Virgin-HMA mixture, showing less ageing occurrence) can also be observed.

5.4 Conclusions

- The incorporation of 50% RAP material into FBMs manufactured by means of a mechanical process and by the addition of zeolites into the bitumen, produced a different rheological

response in the final mixtures to that seen in the FBMs manufactured with the same foaming processes, without RAP material, in Chapter 4.

- As the production temperature increased, and for the same RAP content (i.e. 50%) both Mechanical foamed-RAP and Zeolite-RAP mixtures exhibited an increase in their dynamic shear modulus. The level of blending between the old bitumen from RAP and the soft virgin bitumen, along with the production processes (i.e. mixing), and foaming technology, were the main factors influencing this behaviour.

- Blending analysis comparing the dynamic shear modulus master curves of the Mechanical foamed-RAP and Zeolite-RAP mixtures produced at temperatures of 90, 120 and 160ºC to that of the Virgin HMA suggested that significant blending of the RAP and soft virgin binders occurred in all the Zeolite-RAP mixtures evaluated, and the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC. These results are considered positive, as they suggest that 50% of the virgin materials can be replaced by RAP material, employing production temperatures as low as 90ºC, to produce a mixture with similar characteristics to that of a conventional HMA mixture, generating energy savings, and contributing to the conservation of natural resources.

- The blending analysis comparing the dynamic shear modulus master curve of the Mechanical foamed-RAP mixture manufactured at 160ºC and the HMA-RAP mixture, with that of the Virgin HMA, on the contrary, was not consistent with the previous findings for the other mixtures. These mixtures exhibited high dynamic shear modulus values, similar to those reported for the 100%RAP mixture, suggesting that the behaviour of these mixtures were primarily dominated by the hardened bitumen from RAP, and reflecting the important contributions of other factors to the mixture modulus.

- Statistical analysis of the dynamic shear moduli and phase angle confirmed that the properties of the Zeolite-RAP mixtures manufactured at 90, 120 and 160ºC, and the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC, were not statistical significant from those of the Virgin HMA. On the contrary, the Mechanical foamed-RAP mixture manufactured at 160ºC and the HMA-RAP mixture exhibit statistical significance from the Virgin HMA, indicating that complete blending did not occur in these mixtures.

- Complete blending was initially assumed in order to target a final penetration of the mixtures containing 50% RAP with the two foaming technologies that would equal that of the Virgin mixture. However, different degrees of blending were obtained for all the mixtures with

production temperature, as it could be expected. An accurate determination of the actual level of blending could not be obtained. However, the different degrees of blending can be interpreted from the $|G^*|$ master curves of the mixtures.

- The rheological indexes were able to relate the rheological properties of all the FAM mixtures containing RAP with their performance in terms of cracking. Also, the parameters were able to capture the benefits of a 100% virgin material and the use of low production temperatures to reduce ageing of the mixtures.

- Finally, both parameters ranked the HMA-RAP and the Mechanical foamed-RAP mixtures, manufactured at 160ºC as the most susceptible to cracking.

- FBMs containing RAP material showed a clear change in the rheological parameters (β and γ) and in the Glover-Rowe parameter. The FBM-RAP materials experienced trends associated with age hardening as production temperature increased, indicating short-term ageing or additional RAP-virgin binders blending occurring during mixtures' production.

Chapter 6 : Environmental sensitivity assessment of RAP-foamed bitumen mixtures

This chapter evaluates the influence of an environmental conditioning process that incorporates various processes such as the effects of water infiltration and oxidative ageing on the durability of RAP-FBMs. The conditioning process consisted of initial saturation, prior to placing the FAM samples in a water bath at 60ºC for 6, 15 and 30 days. The effects of this conditioning process on the FAM mixtures was studied through rheology, applying the DMA testing technique to obtain the linear viscoelastic properties (i.e. $|G^*|$ and δ) of these materials before and after the conditioning times.

6.1 Characteristics of the FAM mixtures

For this part of the study, a total of five FAM mixtures were assessed which form part of the set of mixtures that were evaluated in Chapter 5. These mixtures include the virgin HMA mixture—no RAP material- manufactured at 160°C, with no foaming technology (Virgin-HMA), which corresponds also to the materials used to produce lab-RAP, the HMA mixture with 50% RAP material manufactured at 160°C with no foaming technology (HMA-RAP), and several of the RAP-FBM mixtures initially evaluated in Chapter 5. In terms of the RAP-FBM mixtures produced through a laboratory foaming plant, they correspond to those with a final temperature after mixing of 90 and 120ºC, labelled as 'Mechanical foamed-RAP- 90°C' and 'Mechanical foamed-RAP-120°C' respectively. Finally, the selected RAP-FBM manufactured by the addition of zeolites corresponds to the mixture manufactured at 120°C, labelled as 'Zeolite-RAP- 120°C', as it constitutes the main application of this foaming technology (i.e. warm processes). Table 6-1 summarizes the characteristics of these mixtures. Details on the properties of the base materials of these mixtures, the corresponding mix design and production process are found in Chapter 3, Sections 3.1.1 and 3.1.2

Table 6-1 Production temperatures and temperatures of the constituent materials for the FAM mixtures

Mixture Type	T of the Materials (ºC)			Mixing T (ºC)	Foaming technology
	Bitumen T (ºC)	Aggregates T (ºC)	RAP T (ºC)		
Virgin HMA	160	160	-	160	None
HMA-RAP	160	160	160	160	

Mixture Type	T of the Materials (ºC)			Mixing T (ºC)	Foaming technology
	Bitumen T (ºC)	Aggregates T (ºC)	RAP T (ºC)		
Mechanical foamed-RAP-90C	160	110	110	90	Mechanical
Mechanical foamed-RAP-120C	160	140	140	120	foaming
Zeolite-RAP-120C	120	120	120	120	Zeolite

6.2 Environmental conditioning for the RAP-FBMs

From the same SGC specimens of 150 mm in diameter and 90 mm in height that were prepared for the same mixtures evaluated in Chapter 5, and also evaluated here, further cylindrical FAM specimens of 50 mm in height by 12.5 mm in diameter were obtained for this part of the study to be subjected to the environmental conditioning protocol. The initial condition (dry) samples correspond to those specimens tested in Chapter 5. The volumetric properties therefore are the same (Table 5.3).

Three further specimens from each type of mixture were subjected to the environmental conditioning protocol for 6, 15 and 30 days conditioning times. The conditioning protocol consisted of submerging the small cylindrical samples in deionized water under vacuum and under a pressure of 6.7 Mbar for 30 min, as suggested by BS EN 12697-12:2008. Then, the samples were kept submerged in deionized water for another 30 min, after which they were transferred to a water bath at 60°C for the conditioning times. The temperature of 60ºC for the water bath was chosen to investigate whether the addition of moisture at elevated temperatures would produce greater levels of specimen damage. Samples were placed in a mould (Figure 6.1) to avoid deformation during the conditioning process in the water bath. The samples were then removed from the water bath and air-dried for 24 hours prior to testing. Testing of the environmental-conditioned samples was completed within 24 to 36 hours after removal from the water.

Figure 6.1 FAM samples placed in the mould for environmental conditioning

Frequency and temperature sweep tests were conducted on the cylindrical FAM samples for all the mixtures by means of DMA testing to determine the linear viscoelastic material properties using the testing conditions described in Section 3.2.1.

6.3 Initial condition

The average master curves of the dynamic shear modulus, $|G^*|$ of tested specimens for each mixture at a reference temperature of 25ºC in dry conditions (i.e. prior to conditioning) are summarized in Figure 6.2.

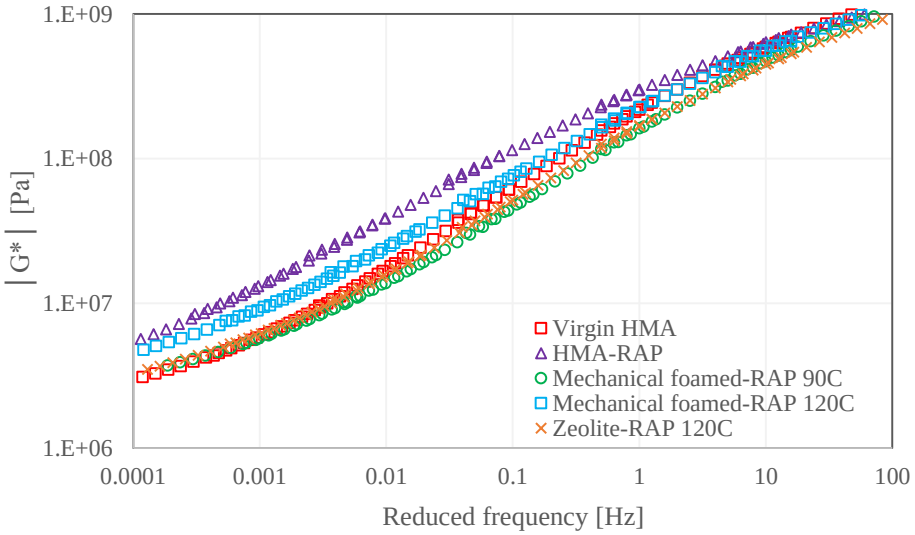


Figure 6.2 Master curves of |*G| for all the RAP-FBM mixtures in dry condition**

Under dry conditions, Figure 6.2 shows that the material HMA-RAP (without foamed bitumen) and the Mechanical foamed-RAP 120ºC exhibit the highest $|G^*|$ values, while the Mechanical foamed-RAP-90ºC and the Zeolite-RAP-120ºC mixtures exhibit the lowest $|G^*|$ values. The latter mixtures exhibit similar $|G^*|$ values between them, and also compared to those of the Virgin HMA. These results imply that the RAP mixtures exhibited a different shift of the $|G^*|$ values with reference to the Virgin-HMA. Indeed, while complete blending was initially assumed, to target a final penetration of the mixtures containing RAP, equal to that of the Virgin HMA, the degree of blending was found to be a function of the production temperature and foaming technology (i.e. particularly mixing) used to manufacture the FAM mixtures, as discussed in Chapter 5.

Figure 6.3 shows the rheological results of the mixtures in the Black space. In this space, all the frequencies and temperatures tested are displayed, corresponding lower temperatures to higher values of $|G^*|$ and higher temperatures to lower values of $|G^*|$. The Mechanical foamed-RAP mixture manufactured at 90ºC and the Zeolite-RAP mixture manufactured at 120ºC show similar black

diagrams, similarly to what has been seen in their $|G^*|$ master curves. An increase in the production temperature for the Mechanical foamed-RAP mixture to 120ºC (Mechanical foamed-RAP 120ºC), increases the elastic response of the material (slightly lower δ values). The HMA-RAP material show high elastic response (low δ values) at low temperatures, due to the increase in stiffness observed in the $|G^*|$ master curve, meaning more susceptibility to fatigue and thermal cracking potential. Furthermore, at high temperatures all the RAP mixtures exhibit a pronounced shift towards lower δ values with respect to the Virgin HMA, indicating the more elastic behaviour due to the presence of the hard bitumen from RAP.

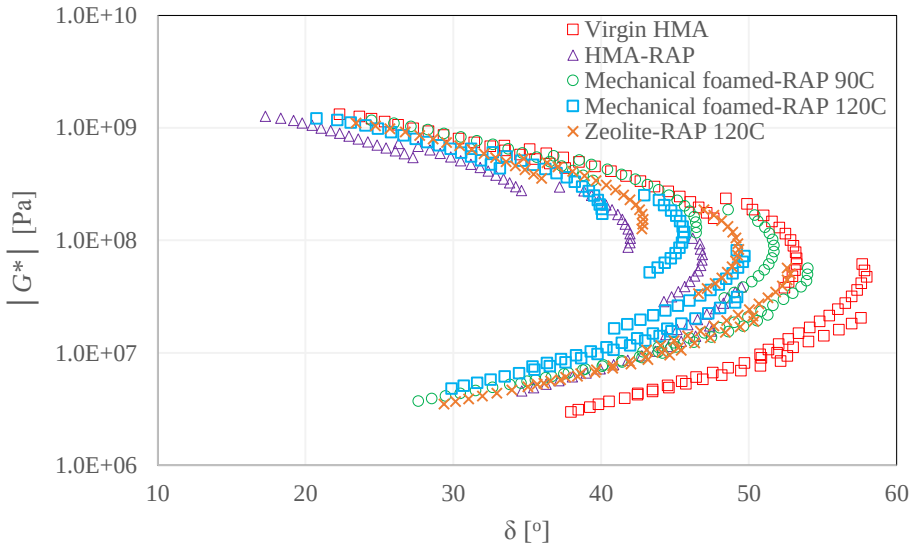


Figure 6.3 Black diagram for all the RAP-FBM mixtures in dry condition

A statistical analysis was also conducted to evaluate the significance of the incorporation of RAP material at different production temperatures and with both foaming technologies on the $|G^*|$ of the mixtures. In the analysis, the $|G^*|$ values at all tested frequencies and temperatures for all the RAP mixtures were compared to those of the Virgin HMA. In general, the statistical analysis shows that at testing temperatures of or below 25ºC (which corresponds to high reduced frequencies in the $|G^*|$ master curves), all the mixtures are statistically similar. The $|G^*|$ values at these testing conditions correspond to the maximum stiffness of the mixtures, which tend to be similar in all the mixtures, having the same mixture design. At testing temperatures of or above 35ºC, the HMA-RAP mixture is statistically different from the Virgin HMA, thus indicating that the $|G^*|$ values of these mixtures are different, so complete blending between the old-virgin binders did not occur. The Mechanical foamed-RAP mixture manufactured at 120ºC shows statistical significance at testing temperatures of or above 45ºC, with respect to the Virgin HMA, meaning that higher production temperatures have an important effect on the $|G^*|$ of the mixtures. Furthermore, the Mechanical foamed-RAP mixture manufactured at 90ºC and the Zeolite-RAP mixture manufactured at 120ºC are statistically the same between them and also compared to the Virgin HMA, at temperatures of or above 35ºC, meaning that

these mixtures are comparable to those of the Virgin HMA. In general, the statistical analysis confirms that the production temperature and foaming technology have an important effect on the rheological response of the mixtures, leading to different stiffening effects with respect to the Virgin HMA, particularly at mixing temperatures of 160ºC.

6.4 Dynamic Mechanical Analysis

The |G*| master curves in Figure 6.4 for the Virgin-HMA mixture after wet conditioning, show an increase in |*G**| values with increasing conditioning times. This behaviour would appear initially to be unusual as it was expected that the presence of moisture in the environmental conditioning would result in damage of the FAM specimens and a reduction in |*G**| values with increasing conditioning times. However, it is important to appreciate that in addition to the effect of moisture-induced damage in this conditioning process, there will be an element of ageing, acting on the FAM specimens while present in the 60°C water bath and, most likely, this could result in a hardening (stiffening) of the FAM specimens. At this temperature in the water bath, it would be possible that the binders present in the FAM mixtures experience oxidative ageing which results in the gradual hardening of the FAM mixtures over the conditioning times.

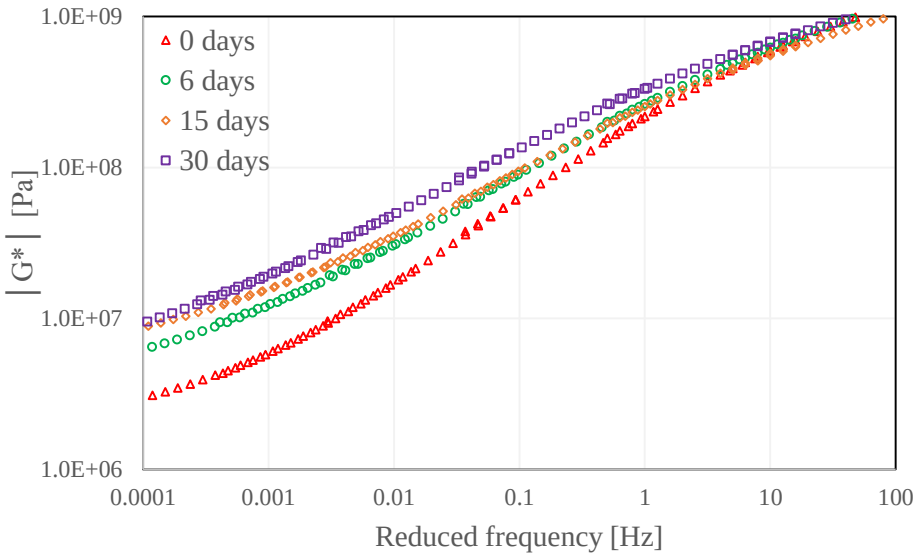


Figure 6.4 Master curve of |G*| for the Virgin HMA

It is likely that all these mechanisms will act in combination with opposite effects in terms of stiffness change and that what is seen in Figure 6.4 is the composite (combined) effect of stiffening and degradation in $|G^*|$. It is therefore also important to compare the responses seen in Figure 6.4 to those seen in Figure 6.5 to get a true representation of the environmental damage effects experience in the RAP-FBM materials. The master curves presented in Figure 6.5 show that the |G*| master curves

of the different FBMs containing RAP for different conditioning times are very similar. Comparing the data in Figure 6.5 to that seen of the Virgin-HMA mixture in Figure 6.4, it can observed that there is a lower increase in the $|G^*|$ values of the RAP-FBM mixtures after the environmental conditioning. It is particularly noticeable that, in most cases, the mixtures exhibited a gradual increase in $|G^*|$ with greater conditioning times. However, this effect was sometimes reversed, as depicted by a slight decrease in $|G^*|$ that occurred in the Zeolite-RAP at 120ºC for example (i.e. 15 days conditioning).

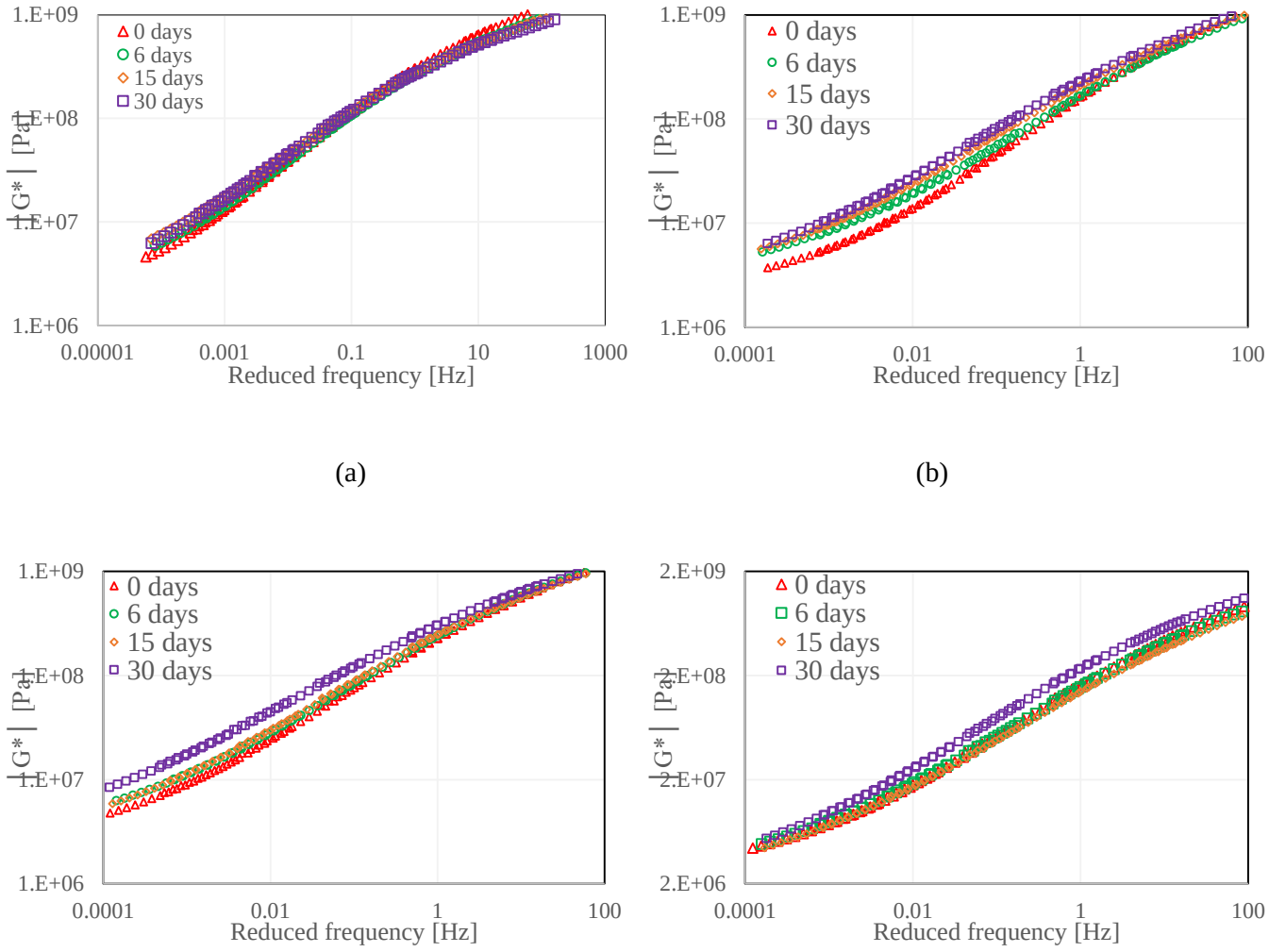
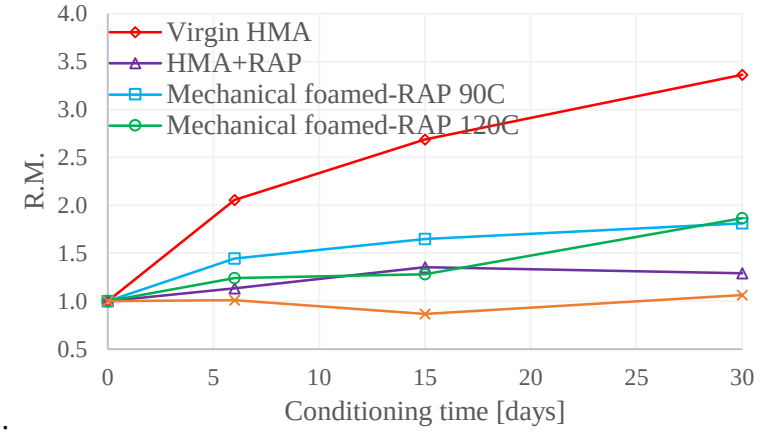


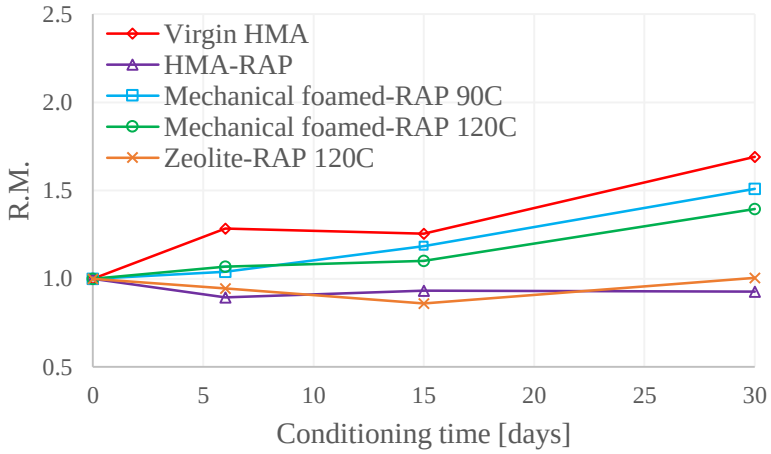
Figure 6.5 Master curve of |G*| for (a) HMA-RAP, (b) Mechanical foamed-RAP 90ºC, (c) Mechanical foamed-RAP 120ºC, and (d) Zeolite-RAP 120ºC

In order to better quantify the actual effect of the environmental conditioning on the $|G^*|$ response of the RAP-FBM materials, the information from the master curves was used to calculate the retained modulus (R.M) ratios (herein defined as the ratio of the average |G*| after each conditioning time divided by the initial |G*| in dry condition) for all the mixtures at two loading frequencies and one

temperature. These results are presented in Figure 6.6 for 0.001Hz (where more changes in the $|G^*|$ were observed), and at a higher frequency value of 0.5Hz, at one temperature of 25ºC



a)



b)

Figure 6.6 R.M. for all the mixtures for different conditioning times at a) 0.001Hz and b) 0.5 Hz

These figures corroborate the behaviour of combined stiffening and degradation effects on the $|G^*|$ values of the mixtures over the different conditioning times. The mixture that exhibited the highest rise in modulus compared to the dry condition for both frequencies was the Virgin-HMA (the R.M. shows an increase in |G*| of 236% at 0.001Hz, and of 69% at 0.5 Hz) both occurring after 30 days conditioning time, while the Zeolite-RAP-120°C exhibited the lowest R.M. values in |G*| for both frequencies, (a reduction of 13% at 0.001 Hz, and a reduction of 14% at 0.5 Hz), both occurring at 15 days conditioning time. One relevant observation from Figure 6.6 is that even though the Zeolite-RAP mixture presented the lowest changes in the values of $|G^*|$, it seems to be the one in which a larger structural deterioration is being produced due to the presence of moisture in the environmental conditioning as its viscoelastic properties are being negatively reduced in some conditioning times.

Remembering that the R.M. values combine the two opposite effects of ageing (stiffening) and moisture damage (degradation) that occur during the environmental conditioning, it is clear that the Zeolite-RAP-120°C showed the lowest resistance to the degradation effects generated by the presence of moisture followed by the HMA-RAP mixture, the two Mechanical foamed RAP-FBM materials (Mechanical foamed-90°C and Mechanical foamed-120°C) and the Virgin HMA.

As an aside and to support the observations above, previous rheological and chemical studies conducted on a virgin bitumen submerged in distilled water for periods of up to nine months, demonstrated that the moisture conditioning process generated changes in the chemistry of the material that could be attributed to oxidative ageing (Figueroa Infante, 2015). Furthermore, other studies on the evaluation of stiffness of asphalt mixtures measured by the ITSM (Indirect Tensile Stiffness Modulus) test after moisture conditioning, where the tested specimens were placed in a water bath at 60ºC for up to 6 hours, followed by a cold water bath at 5ºC for 16 hours and finally transferred to a water bath at 20ºC for at least 2 hours, showed a significant increase in the measured stiffness at 20ºC. In this study, the increase in stiffness after the moisture condition protocol was attributed to binder ageing taking place during the procedure of moisture conditioning (Aljuboryl et al., 2017).

Moreover, results from other studies conducted to evaluate a combined effect of ageing and moisture sensitivity on the stiffness of asphalt mixtures showed that the mixtures exhibited an initial stiffening effect, followed by a decrease in modulus after various combined ageing and moisture sensitivity regimes (Airey et al., 2005). Therefore, it is speculated that the stiffening effects caused by moisture that were observed in this study for the majority of the mixtures are mainly due to changes in the rheological and cohesive properties of the bitumen. However, it is hypothesized that for further conditioning times, the |G*| of the mixtures could reach a maximum value after which a decrease in modulus might start to occur, evidencing the degradation of the adhesive bonds between the fine aggregate particles and the bitumen in these mixtures under the presence of moisture. Furthermore, it is hypothesised that the small size air voids of these FAM materials, and their high total bitumen content, could significantly impact the degree to which moisture can reach both the inner portion of the specimen and the adhesive interfaces of the fine aggregate particles, as already demonstrated by Vasconcelos (2010). Therefore, it is expected that after 30 days of moisture conditioning, water may only have reached a limited portion of the specimen and, consequently, longer conditioning times could change the observed trend in the change of the viscoelastic properties of the mixtures.

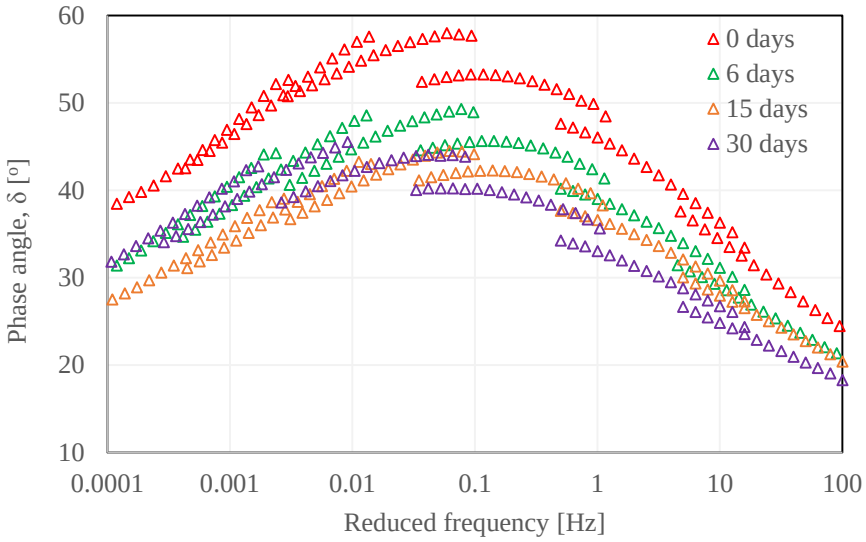
In addition, it is important to mention that these mixtures were manufactured with limestone aggregates, which are well known for their resistance to moisture damage. Therefore, it is possible

that the relative performance of these mixtures under the environmental effects, which include the presence of moisture, is also influenced by the properties of these aggregates.

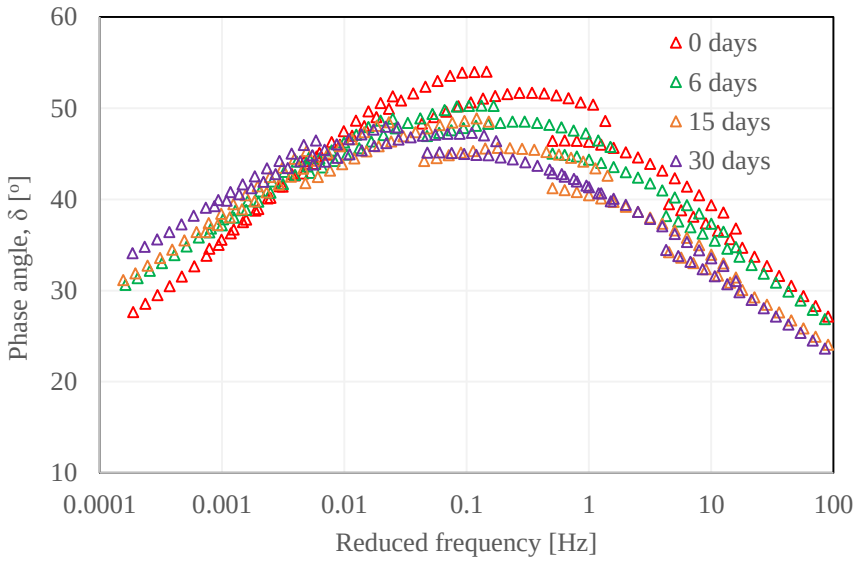
Phase angle master curves for all the mixtures were constructed using the same shift factors as the $|G^*|$ master curves presented previously. The δ master curves for the FAM mixtures containing RAP material are very similar and therefore, the δ master curve for the Virgin HMA and for only one RAP mixture will be presented for conciseness. The δ master curves for the rest of the mixtures can be found in Appendix C.

The δ master curves in Figure 6.7a for the Virgin HMA mixture show a clear reduction of the δ values (more elastic response) with respect to the initial condition, after the environmental conditioning and at all reduced frequencies. With increasing conditioning times this increased elastic response is clearly observed at high-reduced frequencies or low temperatures. For instance, at 10Hz, the δ decreases from 36^o at the initial condition, to 24^o after 30 days conditioning time. However, at low-reduced frequencies or high testing temperatures the δ values at 6 and 30 days conditioning overlap and there is even an increase in δ (more viscous response). At high-reduced frequencies (or low testing temperatures), the behaviour of the base bitumen is rheologically dominant. Therefore, the decrease in δ at these conditions can be associated solely with the ageing of the bitumen present in the FAM specimens while in the 60°C water bath. The unusual behaviour of the δ at the low-reduced frequency conditions (or high temperatures) could be related to the shifting procedure of the master curves, or might be an effect of the frequency during the testing.

In terms of the RAP-FBMs, Figure 6.7b shows that at the initial condition (i.e. 0 days conditioning), the δ values of the Mechanical foamed-RAP mixture manufactured at 120^{o}C are lower compared to those of the Virgin HMA (no RAP material). For instance the Virgin HMA exhibit maximum δ values up to 57^o, whereas the Mechanical foamed-RAP mixture manufactured at 120^{o}C exhibit maximum δ values of 51^o. This reduction in δ, is a result of the increased elastic response of the bitumen present in the RAP material. After the environmental conditioning, the δ exhibits in general a similar behaviour in the RAP-FBMs over the range of frequencies as that observed for the Virgin-HMA, with the δ values after the environmental conditioning being closer to those of its initial condition (i.e. 0 days) in the RAP-FBMs. Therefore, the changes in δ of the RAP-FBMs mixtures after the environmental conditioning process resulted in an increase in the elastic behaviour at high-reduced frequencies or low testing temperatures, and a more viscous response at low-reduced frequencies or high testing temperatures, with conditioning time.



a)



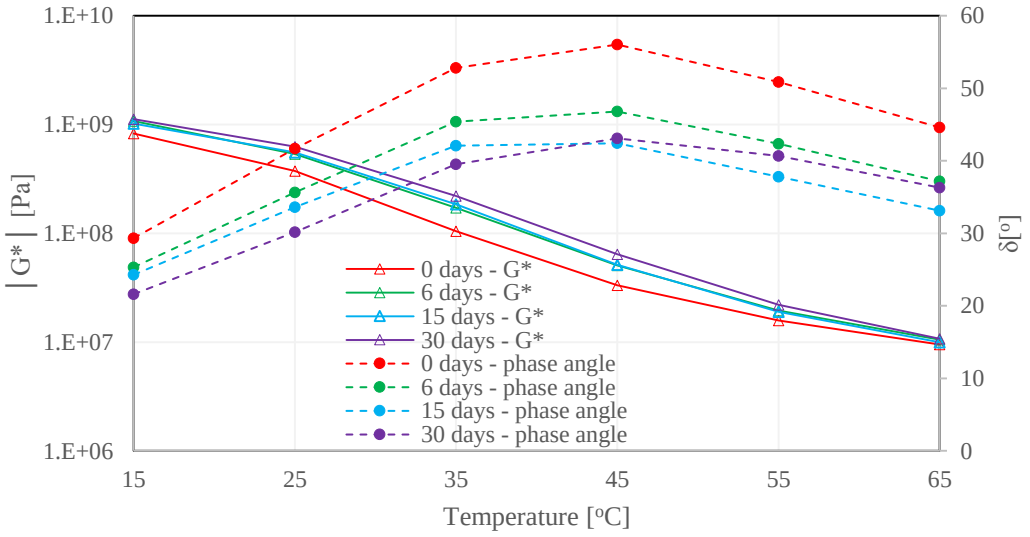
b)

Figure 6.7 δ master curve for a) Virgin HMA, and b) Mechanical foamed-RAP at 90ºC mixture

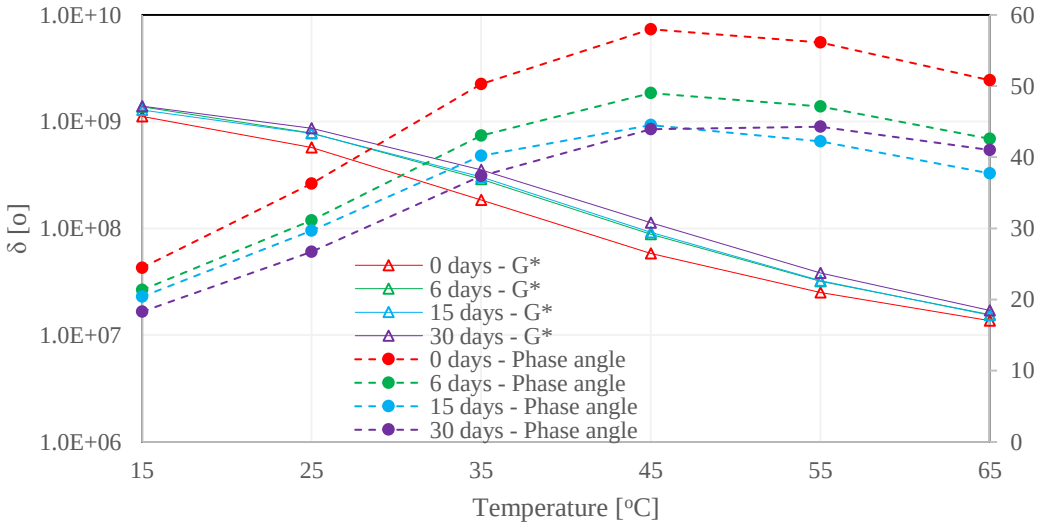
The discontinuous shifting of the δ data to form the master curves in Figure 6.7 reflects that the materials does not exhibit a thermorheologically simple behaviour. Therefore, the behaviour of $|G^*|$ and δ after the environmental conditioning were evaluated also by means of isochronal plots at two constant frequencies of 3.162Hz and 10Hz. Figure 6.8 and Figure 6.9 present the isochronal plots for the Virgin HMA and for one RAP mixture (e.g. Mechanical foamed-RAP at 120ºC) as an example, as similar behaviour was observed within the RAP mixtures.

The isochronal plots for the Virgin HMA at the two frequencies show a similar increase in $|G^*|$ values with conditioning time. There is also a regular decrease in δ with conditioning time at both

frequencies, which is more significant between 15ºC to 45ºC, after which there is an increase in δ towards a more viscous behaviour at 30 days conditioning. This means that the relative increase in viscous response changes not only for loading time, as seen in the δ master curves, but also for temperature.



a)

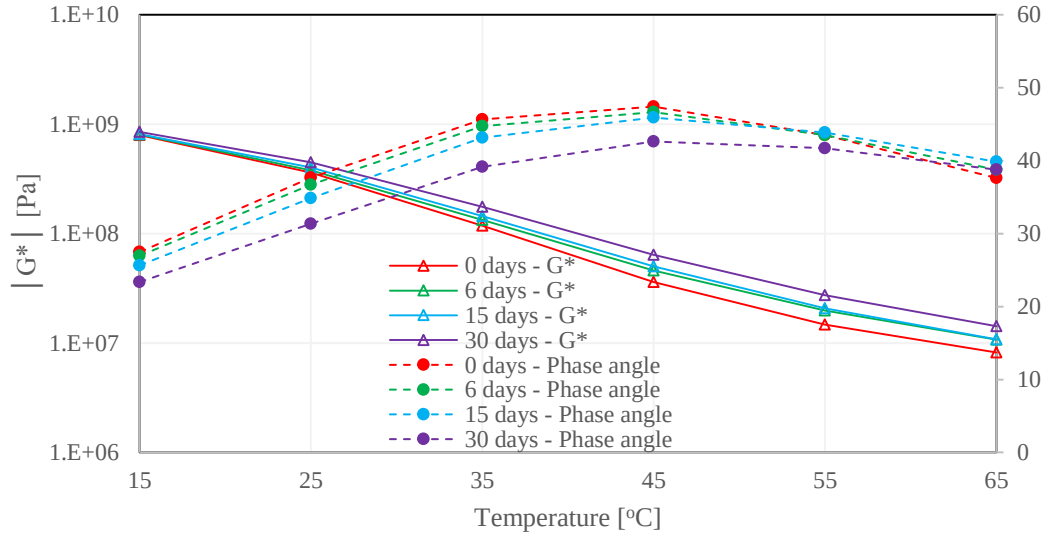


b)

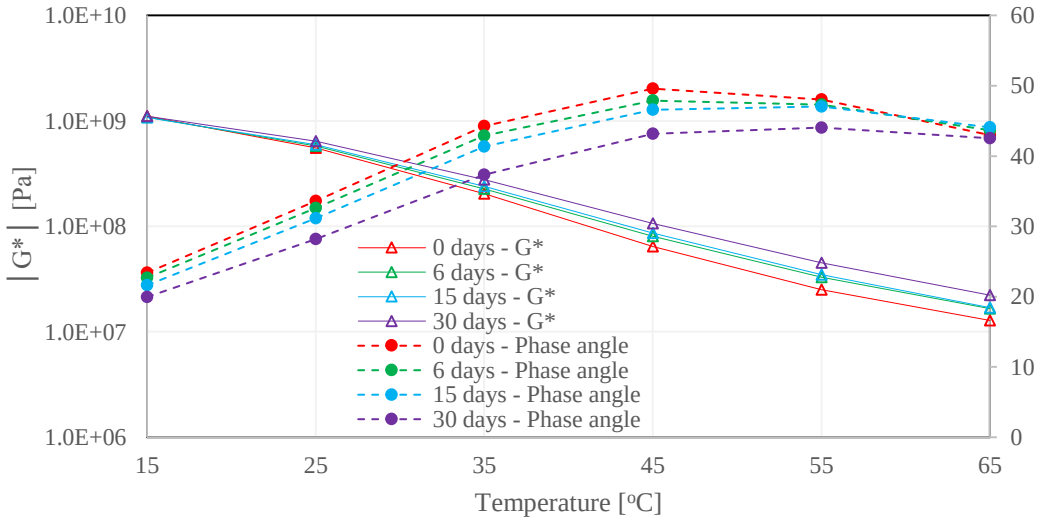
Figure 6.8 $|G^*|$ Isochronal plots at a) 3.162Hz and b) 10Hz for the Virgin HMA

The isochronal plots for the Mechanical foamed-RAP at 120ºC show a lower increase in $|G^*|$ with conditioning time, with respect to that seen for the Virgin HMA, and this increase becomes more significant at temperatures above 25ºC. At temperatures below 25ºC, there is a slight reduction in the magnitude of increase in $|G^*|$. The different rheological behaviour of the two FAM groups results in the increase of $|G^*|$ being greater for the Virgin HMA compared to the Mechanical foamed-RAP at

120ºC over the range of temperatures. Furthermore, there is a similar decrease in δ towards a more elastic behaviour for the Mechanical foamed-RAP at 120ºC between 15ºC to 45ºC, but this decrease is lower compared to that observed for the Virgin HMA, and after 45ºC the δ values of the Mechanical foamed-RAP 120ºC mixture with conditioning time tend to be very similar or in some cases they overlap, which was also observed in the δ master curve.



a)



b)

Figure 6.9 $|G^*|$ Isochronal plots at a) 3.162Hz and b) 10Hz for the Mechanical foamed-RAP 120ºC

6.5 Statistical analysis on $|G^*|$ and δ

In order to quantify the significance of the conditioning times during the environmental conditioning on the rheological properties (i.e. $|G^*|$ and δ) of both foaming RAP groups, the $|G^*|$ and δ raw data obtained from the frequency sweep and temperature tests were analysed statistically. The analysis was conducted similarly to that in Chapter 5, Section 5.2.4, using independent sample t-tests with a confidence interval of 95%. In general the analysis consisted of comparing the $|G^*|$ and δ raw data of the samples tested at each conditioning time to that of the initial condition, for each mixture. Table 6-2, presents a summary of the results at two selected frequencies and at all testing temperatures. In the analysis the null hypothesis is that the means of the samples tested at the initial condition to those of the samples tested after 6, 15 and 30 days conditioning times are not significantly different (e.g. $|G^*|$ at the initial condition for the Mechanical foamed-RAP mixture manufactured at 90ºC and the $|G^*|$after 30 days conditioning time for the same mixture, are not significantly different). A *p*-value equal to or less than 0.05 are statistically significant (green shaded values in Table 6-2). Otherwise, results yielding a *p*-value above 0.05 are considered not statistically significant. Appendix B contains the detailed procedure followed to perform the statistical analysis.

The statistical analysis results show that in general the effects of the environmental conditioning on the rheological properties of the RAP and virgin materials are different for each type of mixture, with conditioning time, and over the frequencies. For instance, for the Virgin HMA, the $|G^*|$ and δ values after 6, 15 and 30 conditioning days in the environmental conditioning are significantly different from its initial condition, and these differences are more significant at temperatures of or above 35ºC (which correspond to low reduced frequencies in the master curves). Meaning that changes in the rheological response of Virgin HMA due to the environmental conditioning, started being noticeable after 6 days conditioning. The HMA-RAP mixture on the contrary, show little statistical significance from its initial condition for all conditioning times, meaning that the rheological properties of this mixture were not significantly affected by the environmental conditioning effects, as was observed in the $|G^*|$ master curves. Furthermore, the Mechanical foamed-RAP mixture manufactured at 90ºC show statistical significance from its initial condition after all conditioning times (i.e. 6-30 days), and this significance becomes higher with testing temperature (corresponding to low reduced frequencies in the master curves). The Mechanical foamed-RAP mixture manufactured at 120ºC, shows statistically significance from the initial condition after 6 and 15 days conditioning only for temperatures of or above 45ºC. After 30 days conditioning, the mixture shows statistically significance from its initial condition, meaning that after 30 days conditioning in the water bath at 60ºC, the rheological properties of this mixture have changed from its initial (dry) condition. In terms of the Zeolite-RAP mixture, this mixture shows little

statistical significance at all conditioning times, from its initial condition, meaning that the properties of this mixture remained almost the same after the environmental conditioning process.

In terms of the δ, the statistical analysis showed that the δ values after all conditioning times are significantly different from those at the initial condition for the Virgin HMA, meaning that both the stiffness and relaxation capability of this mixture were affected similarly by the environmental conditioning. However for the RAP mixtures, the δ values after 6 days conditioning are statistically the same to those of their corresponding initial condition. After 15 days conditioning the δ values for the Mechanical foamed-RAP mixtures show statistically significance at some temperatures. After 30 days conditioning, the δ values are statistical different from their initial condition for the Mechanical foamed-RAP 120ºC mixture and the Zeolite-RAP mixture, meaning that the effects of the environmental conditioning on the δ of these mixtures occurs later in time (i.e. after longer conditioning times). The statistical analysis for the δ of the Mechanical foamed-RAP mixture manufactured at 90ºC shows little statistical significance at all conditioning times.

In summary, the statistical analysis corroborates what has been observed previously from the master curves and retained modulus results, that the Virgin HMA and the Mechanical foamed-RAP mixtures presented large or more noticeable differences in their rheological response after the environmental conditioning. The Zeolite-RAP mixture and the HMA-RAP material were the mixtures that exhibited lower changes in their rheological properties after conditioning in the water bath at 60ºC.

Table 6-2 Statistical analysis Dynamic Shear Modulus – Environmental conditioning

Mixture	T[ºC]	*p*-value							
		0 - 6 days		0 - 15 days		0 - 30 days		15 - 30 days	
		3.162Hz	10Hz	3.162Hz	10Hz	3.162Hz	10Hz	3.162Hz	10Hz
Virgin HMA	15	0.950	0.402	0.079	0.006	0.295	0.345	0.016	0.007
	25	0.250	0.480	0.679	0.569	0.032	0.071	0.003	0.003
	35	0.045	0.075	0.080	0.185	0.005	0.007	0.004	0.009
	45	0.007	0.006	0.005	0.007	0.001	0.001	0.002	0.001
	55	0.003	0.002	0.002	0.001	0.001	0.001	0.413	0.207
	65	0.007	0.004	0.004	0.003	0.000	0.000	0.925	0.628
HMA-RAP	15	0.003	0.001	0.004	0.002	0.001	0.000	0.419	0.245
	25	0.037	0.023	0.075	0.030	0.025	0.009	0.117	0.117
	35	0.420	0.347	0.308	0.778	0.458	0.190	0.141	0.153
	45	0.877	0.712	0.116	0.130	0.383	0.593	0.158	0.160
	55	0.581	0.649	0.068	0.061	0.135	0.145	0.163	0.173
	65	0.162	0.215	0.072	0.062	0.146	0.123	0.142	0.170
Mechanical foamed-RAP 90ºC	15	0.254	0.187	0.770	0.465	0.705	0.396	0.614	0.467
	25	0.914	0.637	0.010	0.091	0.017	0.620	0.190	0.452
	35	0.217	0.335	0.000	0.000	0.006	0.003	0.120	0.073
	45	0.048	0.057	0.000	0.000	0.007	0.003	0.129	0.074
	55	0.034	0.031	0.000	0.000	0.012	0.006	0.172	0.099
	65	0.027	0.019	0.001	0.001	0.031	0.016	0.312	0.201

Mixture	T[ºC]	*p*-value							
		0 - 6 days		0 - 15 days		0 - 30 days		15 - 30 days	
		3.162Hz	10Hz	3.162Hz	10Hz	3.162Hz	10Hz	3.162Hz	10Hz
Mechanical foamed-RAP 120ºC	15	0.819	0.620	0.795	0.182	0.137	0.925	0.195	0.300
	25	0.448	0.516	0.295	0.415	0.018	0.027	0.088	0.113
	35	0.179	0.170	0.119	0.147	0.005	0.007	0.037	0.053
	45	0.035	0.030	0.028	0.024	0.001	0.001	0.014	0.020
	55	0.019	0.019	0.012	0.009	0.000	0.000	0.005	0.006
	65	0.013	0.013	0.007	0.004	0.001	0.000	0.006	0.003
Zeolite-RAP 120ºC	15	0.107	0.047	0.000	0.000	0.007	0.004	0.320	0.721
	25	0.439	0.200	0.002	0.000	0.192	0.029	0.031	0.134
	35	0.723	0.912	0.317	0.051	0.092	0.283	0.034	0.037
	45	0.233	0.179	0.230	0.252	0.030	0.027	0.119	0.084
	55	0.246	0.172	0.207	0.135	0.054	0.034	0.276	0.187
	65	0.438	0.295	0.521	0.288	0.208	0.092	0.579	0.429

Table 6-3 Statistical analysis Phase angle – Environmental conditioning

Mixture	T[°C]	*p*-value							
		0 - 6 days		0 - 15 days		0 - 30 days		15 - 30 days	
		3.162Hz	10Hz	3.162Hz	10Hz	3.162Hz	10Hz	3.162Hz	10Hz
Virgin HMA	15	0.038	0.099	0.055	0.112	0.012	0.033	0.148	0.167
	25	0.040	0.058	0.037	0.063	0.011	0.020	0.092	0.080
	35	0.047	0.040	0.028	0.033	0.014	0.015	0.136	0.060
	45	0.024	0.024	0.012	0.012	0.013	0.010	0.762	0.726
	55	0.010	0.012	0.004	0.006	0.005	0.006	0.405	0.518
	65	0.016	0.011	0.012	0.006	0.016	0.007	0.412	0.385
HMA-RAP	15	0.937	0.876	0.866	0.375	0.043	0.098	0.022	0.016
	25	0.575	0.601	0.287	0.343	0.014	0.008	0.039	0.014
	35	0.713	0.315	0.328	0.095	0.027	0.004	0.168	0.075
	45	0.911	0.411	0.733	0.157	0.320	0.017	0.683	0.337
	55	0.329	0.761	0.285	0.856	0.082	0.885	0.742	0.948
	65	0.148	0.197	0.057	0.117	0.005	0.013	0.534	0.655
Mechanical foamed-RAP 90°C	15	0.442	0.716	0.008	0.015	0.798	0.482	0.375	0.417
	25	0.129	0.156	0.012	0.002	0.091	0.546	0.517	0.420
	35	0.104	0.039	0.044	0.016	0.105	0.062	0.602	0.371
	45	0.303	0.111	0.131	0.053	0.270	0.107	0.971	0.540
	55	0.892	0.397	0.755	0.231	0.743	0.466	0.523	0.888
	65	0.440	0.908	0.193	0.666	0.189	0.446	0.320	0.474

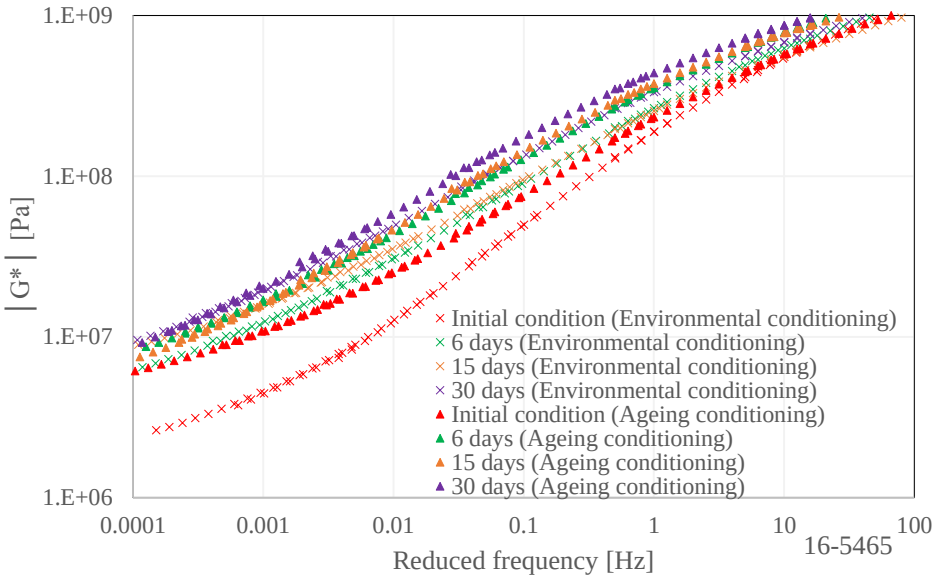
Mixture	T[ºC]	*p*-value							
		0 - 6 days		0 - 15 days		0 - 30 days		15 - 30 days	
		3.162Hz	10Hz	3.162Hz	10Hz	3.162Hz	10Hz	3.162Hz	10Hz
Mechanical foamed-RAP 120ºC	15	0.446	0.506	0.061	0.082	0.008	0.007	0.053	0.072
	25	0.334	0.236	0.026	0.028	0.004	0.004	0.013	0.024
	35	0.428	0.145	0.060	0.019	0.003	0.002	0.001	0.004
	45	0.526	0.140	0.231	0.026	0.014	0.003	0.002	0.001
	55	0.979	0.334	0.654	0.211	0.190	0.013	0.070	0.010
	65	0.079	0.345	0.003	0.086	0.404	0.716	0.428	0.230
Zeolite-RAP 120ºC	15	0.937	0.876	0.866	0.375	0.043	0.098	0.022	0.016
	25	0.575	0.601	0.287	0.343	0.014	0.008	0.039	0.014
	35	0.713	0.315	0.328	0.095	0.027	0.004	0.168	0.075
	45	0.911	0.411	0.733	0.157	0.320	0.017	0.683	0.337
	55	0.329	0.761	0.285	0.856	0.082	0.885	0.742	0.948
	65	0.148	0.197	0.057	0.117	0.005	0.013	0.534	0.655

6.6 Combined laboratory ageing and moisture effects on the $|G^*|$ master curves

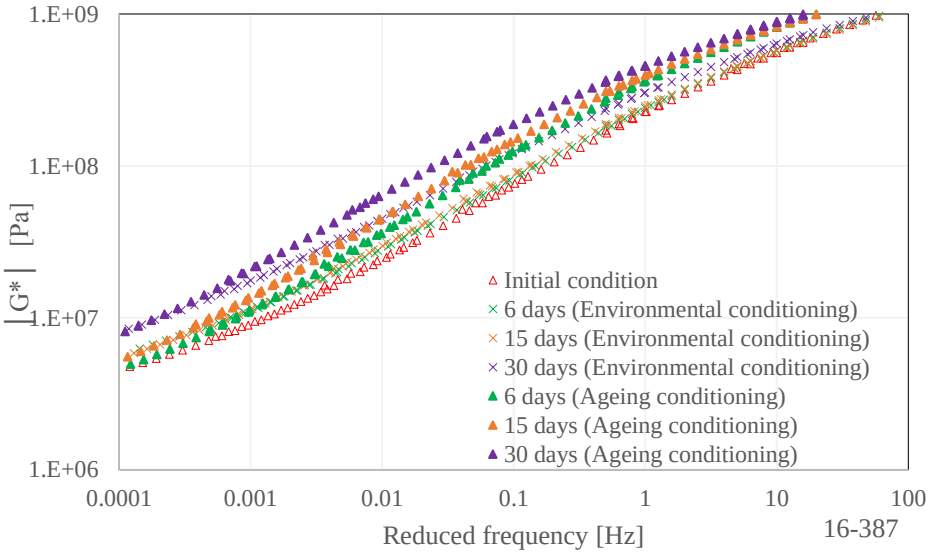
With the objective of isolating the effects of ageing from the combined effects of hardening and degradation observed in the environmental conditioning process, on the $|G^*|$ of the mixtures, the FAM samples were subjected to oven ageing at 60ºC (i.e. the same temperature used in the environmental conditioning process), and for the same conditioning times (i.e. 6, 15 and 30 days). The samples were placed in the same mould as in the environmental conditioning process to avoid deformation during the ageing conditioning process, and also to maintain the same conditions. After ageing conditioning, samples were tested in the DMA test using the same testing conditions described in Section 3.2.1.

Three FAM specimens for each mixture were tested for each conditioning time. As for the environmental conditioning process, these FAM specimens were obtained from the cylindrical FAM samples that were cored from the SGC specimens prepared for the corresponding mixtures that were also evaluated in Chapter 5. However, for this part of the study a new Virgin-HMA mixture was manufactured due to a shortage of FAM samples for this mixture. This mixture was manufactured using the virgin bitumen from the same source to that used for the production of the Virgin-HMA in Section 6.4, however the new bitumen is 20% stiffer (58 pen and 48ºC softening point). Therefore for this mixture, the initial condition for both conditioning processes is not the same. For the RAP mixtures, the initial condition corresponds to the same sets of specimens for the environmental conditioning.

The $|G^*|$ master curves for the environmental and laboratory ageing conditioning processes were combined in one plot for each mixture, in order to evaluate the individual contribution of each conditioning process in the $|G^*|$ of the mixtures. These plots are presented in Figure 6.10 for the Virgin HMA and for the Mechanical foamed-RAP 120ºC. Appendix D contains the curves for the rest of the mixtures. The initial dry (0 days) condition is the same for both conditioning in all the mixtures, except for that of the Virgin HMA mixture since as explained previously, a new Virgin-HMA was manufactured to obtained FAM testing samples of this mixture for the ageing conditioning process.



a)



b)

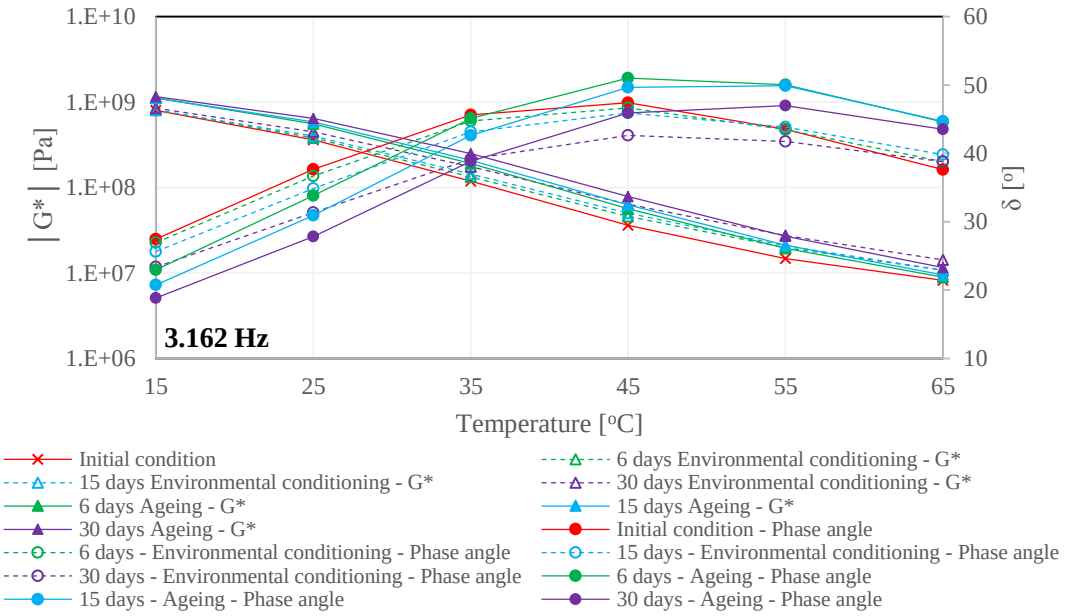
Figure 6.10 $|G^*|$ master curves for a) Virgin HMA and b) Mechanical foamed-RAP 120ºC

These figures corroborate that in both conditioning processes there is a generalized increase in $|G^*|$ of the Virgin HMA and the RAP-FBMs with conditioning time. From a visual inspection of these plots, it can be seen that some curves overlap after 6 and 15 days conditioning times in both conditioning processes. However, after 30 days conditioning in the ageing conditioning process (i.e. no presence of water) the mixtures exhibit the highest $|G^*|$ values with respect to those of the ageing conditioning at 6 and 15 days, and those of the environmental conditioning process at all conditioning times. These results suggest that the ageing conditioning of the virgin and RAP-FBM

materials results in greater stiffness increase compared to ageing-moisture in the environmental conditioning. Indeed with water conditioning either less ageing but also the negative effect of moisture conditioning was observed.

The isochronal plots in Figure 6.11 and Figure 6.12 for two RAP mixtures show in more detail the two behavioural trends observed in the $|G^*|$ in both conditioning processes. For instance, the $|G^*|$ isochronal plots for the Mechanical foamed-RAP 120ºC mixture show an increase in $|G^*|$ in both conditioning processes over the temperature domain, with the relative hardening effects caused by the pure ageing conditioning being higher than those observed in the environmental conditioning process. For the Zeolite-RAP mixture in Figure 6.12, the $|G^*|$ isochronal plot show less ageing as depicted by lower changes in $|G^*|$, but also a negative effect after environmental conditioning. These results suggest that the age-hardening effects are different in each type of mixture. It could be possible that the different manufacturing processes (i.e. particularly mixing), result in mixtures with different aggregate interactions and mobilization of bitumen in the mixture (which was in fact seen from the $|G^*|$ master curves in the initial condition), and hence performance in the mixtures.

In terms of the δ, the δ isochronal plot for both mixtures show a decrease in the δ values with reference to the initial condition after both conditioning processes between 15 and 35ºC, after which there is an increase in the δ values (more viscous response) for the ageing conditioning process. At low temperatures, the rheological behaviour of the bitumen present in the FAM mixtures is dominant, therefore the decrease in δ values is associated to the age-hardening effects experienced in the bitumen during both conditioning processes which results in a more elastic response for the pure ageing conditioning. This unusual increase in δ values at high temperatures after the ageing conditioning, could be an effect of changes occurring in the material at these conditions during the DMA testing, which are not well understood.



a)

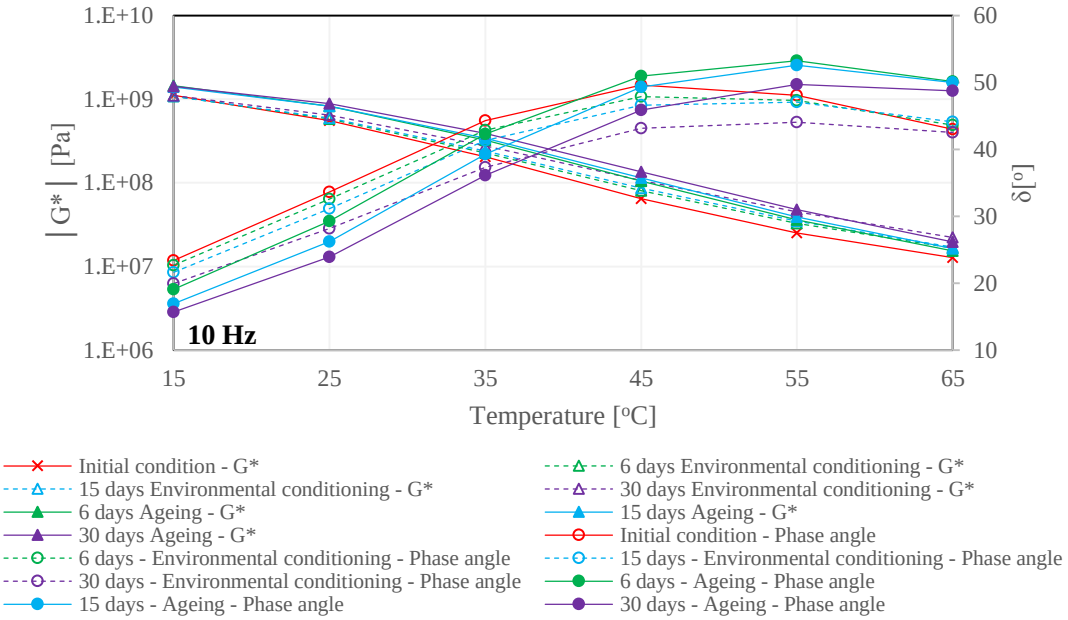
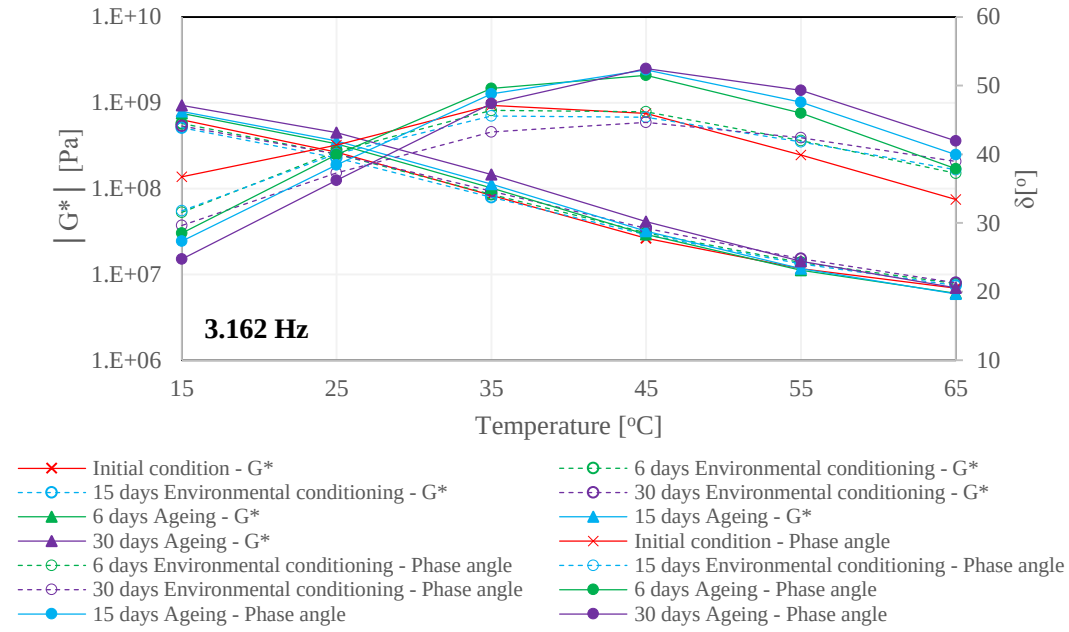
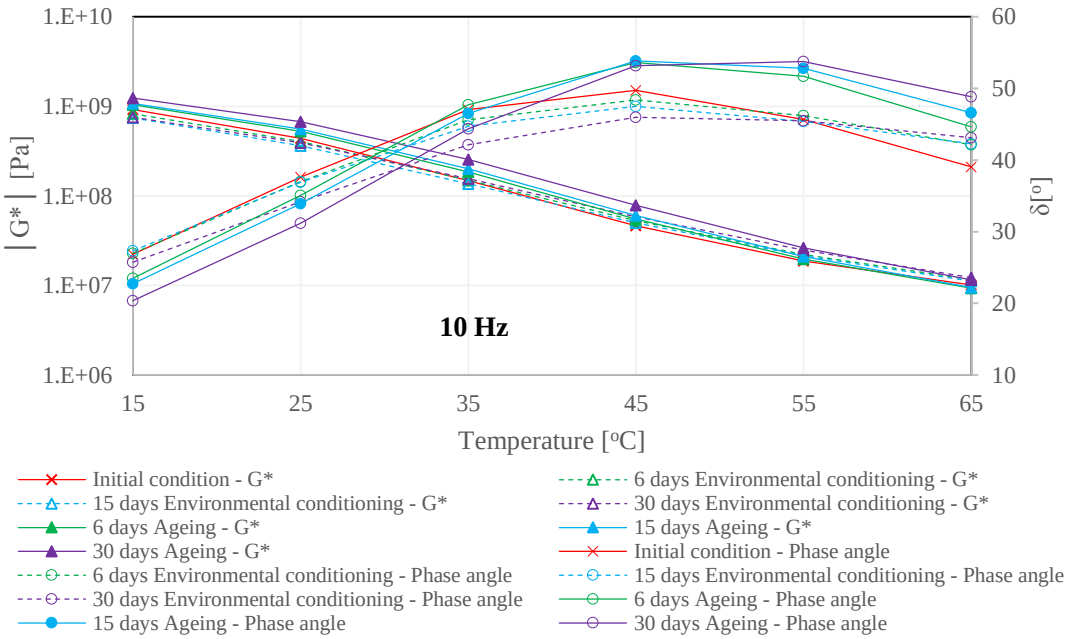


Figure 6.11 $|G^*|$ and δ isochronal plots for Mechanical foamed-RAP 120°C mixture at a) 3.162Hz and b) 10Hz

b)



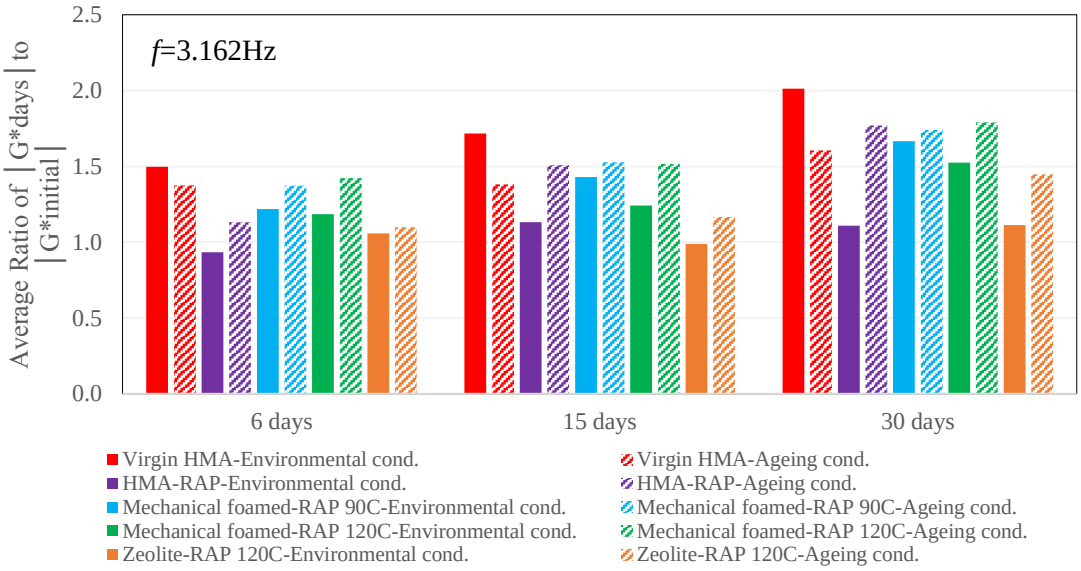
a)



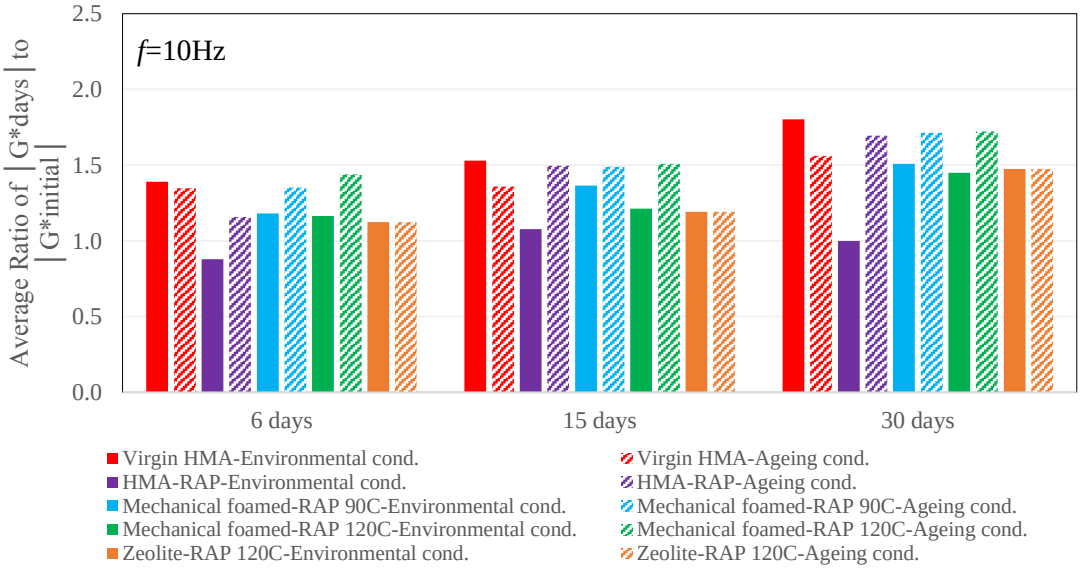
b)

Figure 6.12 $|G^*|$ and δ isochronal plots for Zeolite-RAP 120ºC mixture at a) 3.162Hz and b) 10Hz

With the objective of better quantifying the actual effect of each conditioning process on the $|G^*|$ of the fine RAP-FBMs, dynamic shear modulus ratios were calculated using the $|G^*|$ isochronal plots data, comparing each mixture to its respective initial condition value for both conditioning processes.



a)



b)

Figure 6.13 dynamic shear modulus ratios for all the mixtures at (a) 3.162Hz, and (b) 10Hz

Data in Figure 6.13 corroborates that in both conditioning processes a generalized increase in the $|G^*|$ of the mixtures was observed. These results are understandable for the pure ageing conditioning process. However, for the environmental conditioning process these results partially confirm that ageing of the bitumen while present in the water bath (60ºC) is occurring generating an increase in the stiffness of the FAM mixtures. It is likely that during the environmental conditioning

process, the hardening effects in the bitumen due to oxidative ageing and the degradation of the adhesive bonds between the bitumen and aggregates due to the presence of moisture are occurring simultaneously with the age hardening effects being the dominant effect in the FAM mixtures. This result is also supported by the fact that with the exception of the Virgin HMA, pure ageing conditioning results in greater stiffness increase compared to the environmental conditioning process, in all the mixtures. The greater increase in $|G^*|$ generated after the environmental conditioning process compared to that observed after the ageing conditioning for the Virgin HMA, is a consequence of the stiffer bitumen present in the Virgin HMA material tested for the conditioning process.

In terms of the type of mixtures, the Mechanical foamed-RAP mixtures show higher ratios and larger differences with conditioning time than the Zeolite-RAP mixtures, in both conditioning processes. For instance, on average, the ratios of the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC are 37 and 26% higher than that of the Zeolite-RAP mixture in the environmental conditioning process. In the ageing conditioning, the ratios of the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC are 25 and 27% higher with reference to the Zeolite-RAP mixture. This implies that the ageing-related hardening effects occur in a different extent for the different mixes. The differences in air void could also be contributing to some of the changes in stiffness observed.

At a higher frequency of 10Hz, the results are similar to those reported at 3.162Hz. With the exception of the Virgin HMA (as explained previously), the hardening effects after ageing conditioning were larger than those reported after the environmental conditioning. However, at 10Hz the Zeolite-RAP mixture exhibits consistently similar average ratios in both conditioning processes, and over the conditioning days. These results suggest that the ageing-related hardening effects experienced by the Zeolite-RAP mixture in both conditioning processes were the same (i.e. no moisture damage).

Using the data in Figure 6.13a, the ratios of $|G^*|$ in the environmental conditioning process to that of the $|G^*|$ in the ageing conditioning process for all the mixtures were calculated and compared to their initial stiffness as illustrated in Figure 6.14. An equivalent figure at 10Hz was also constructed, showing the same trend therefore only the plot at 3.162Hz is presented here for conciseness. This figure shows that the higher the initial stiffness of the mixtures, the lower the ageing ratios. For the Virgin HMA, the relative position in this figure is not seen, due to the difference in the stiffness of the binder tested in both conditioning processes. However, in the ideal case were the same bitumen grade was present in the FAM specimens in both conditioning processes, the Virgin HMA would be

expected to be in the area of the plot between the Mechanical foamed-RAP mixture manufactured at 90 and 120ºC.

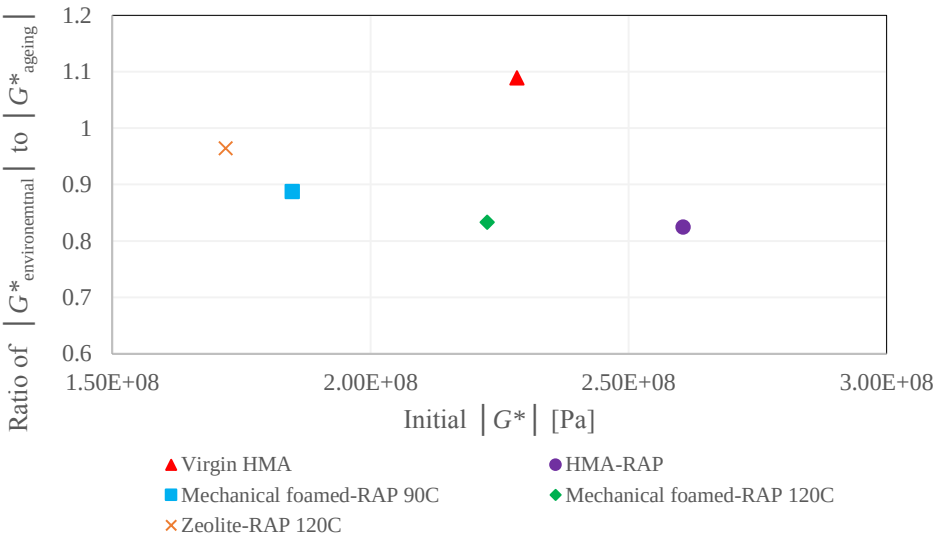


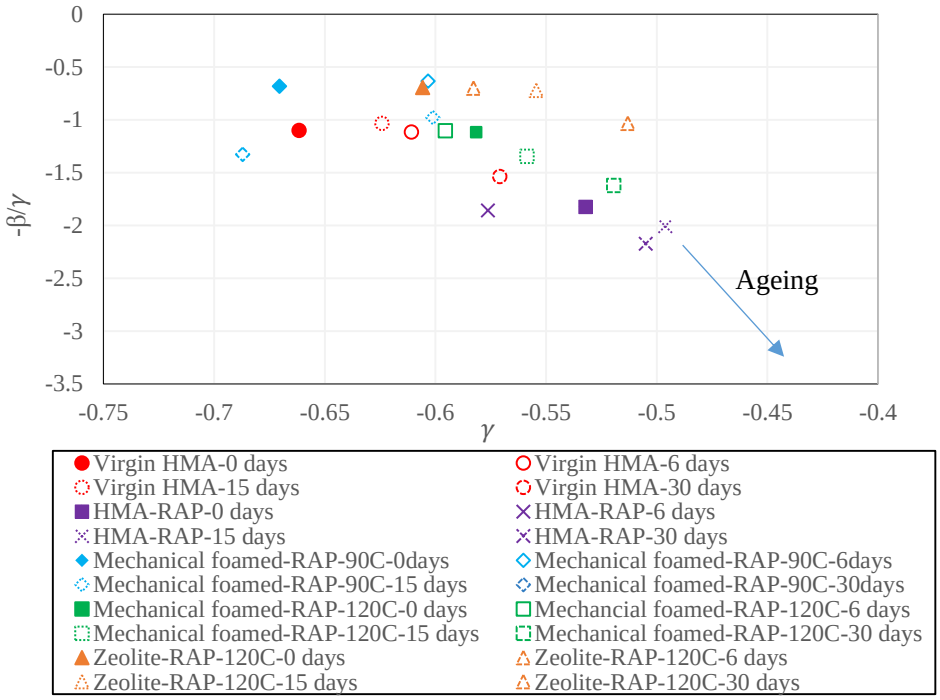
Figure 6.14 Dynamic shear modulus ratios and initial stiffness of the mixtures

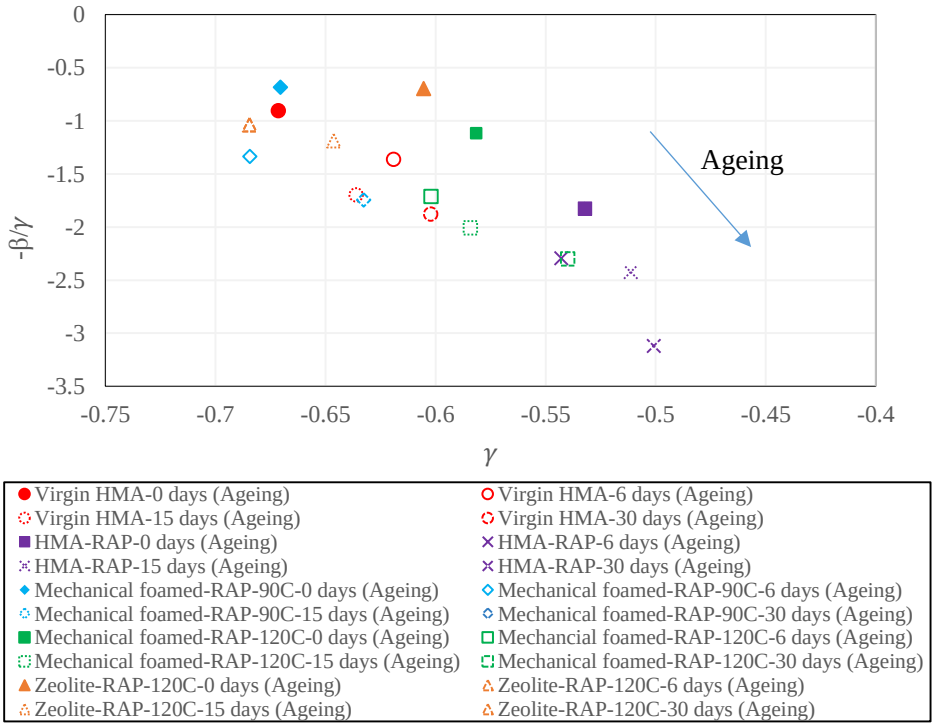
6.7 Rheological indices

In order to support the rheological analysis two indicators that have been proposed in the literature were used: relaxation spectra shape parameters Mensching et al. (2016) and Glover-Rowe (Rowe et al. 2016), and are presented in this section.

6.7.1 Relaxation spectra shape parameters

As described in Chapter 2, Section 2.5.5, Mensching et al. (2016) discussed the physical significance of the fitting parameters (β and γ) of the $|G^*|$ master curve by means of the standard sigmoidal function , as materials age. Thus, with this approach, and after the $|G^*|$ master curves were constructed, the data was fitted using the sigmoidal function (Equation 20) and the corresponding β and γ fitting parameters were determined for each mixture. Figure shows the cross-plot of the – β/γ and γ values for each conditioning process (i.e. environmental conditioning in Figure 6.15 a, and pure ageing conditioning in Figure 6.15b) for the two groups of FAM foaming mixtures with RAP material.





b)

Figure 6.15 Crossover frequency parameter vs. relaxation spectra for virgin and FAM mixtures containing RAP and different foaming technologies after a) environmental conditioning and b) ageing conditioning

In general, although not clear in some cases, this analysis shows that the response of all mixtures with conditioning time moves from the upper left to the lower right in this plot, to areas where more age hardening is occurring. In other words, when the mixtures are subjected to the conditioning processes, the slope of the $|G^*|$ master curves, which is quantified through the γ parameter, exhibit higher values (lower absolute values), reflecting a flattening effect of the master curve which are related to more hardening in the mixtures. Furthermore, the parameter β, which controls the inflection point position of this flattening effect (Mensching et a. 2016), is also moving downwards in these plots. Thus indicating, and as was also observed from the previous rheological results, that after both conditioning processes (Figure 6.15a, and Figure 6.15b) the mixtures experienced an overall hardness.

Furthermore, using the same data from the analysis, the γ parameter was plotted against the conditioning times in Figure 6.16 to assess in more detail the general trend that each mixture exhibits with this variable. In this Figure, it can be seen that at the initial condition, all the mixtures exhibit a relative position with the Mechanical foamed-RAP at 90^{o}C showing the highest γ values, followed by the Virgin HMA, the Zeolite-RAP at 120^{o}C, the Mechanical foamed-RAP at 120^{o}C and the HMA-RAP mixture. The increase in γ values (lower absolute values), correspond to the increase in stiffness of the mixtures. When the mixtures are exposed to the environmental and pure ageing conditioning processes, and rheological changes start occurring, the γ values of each mixture exhibit a relative decrease, which corresponds to a flattening of the mater curve a with respect to their initial condition over the conditioning times, as a result of the age hardening occurring in the mixtures, as explained previously. The response of the mixtures after the pure-ageing conditioning is as could be expected, however for the environmental conditioning, these results validate that ageing is effectively occurring while the samples are placed in the water bath at 60^{o}C for different times. It is also interesting to see that after 30 days conditioning, the relative position of each mixture is different to that observed at the initial condition (with the exception of the HMA-RAP mixture which exhibits the lowest γ values at all conditioning times), suggesting that the age-related hardening effects after both conditioning processes occurring in each mixture are a function of the material as well (i.e. foaming technology).

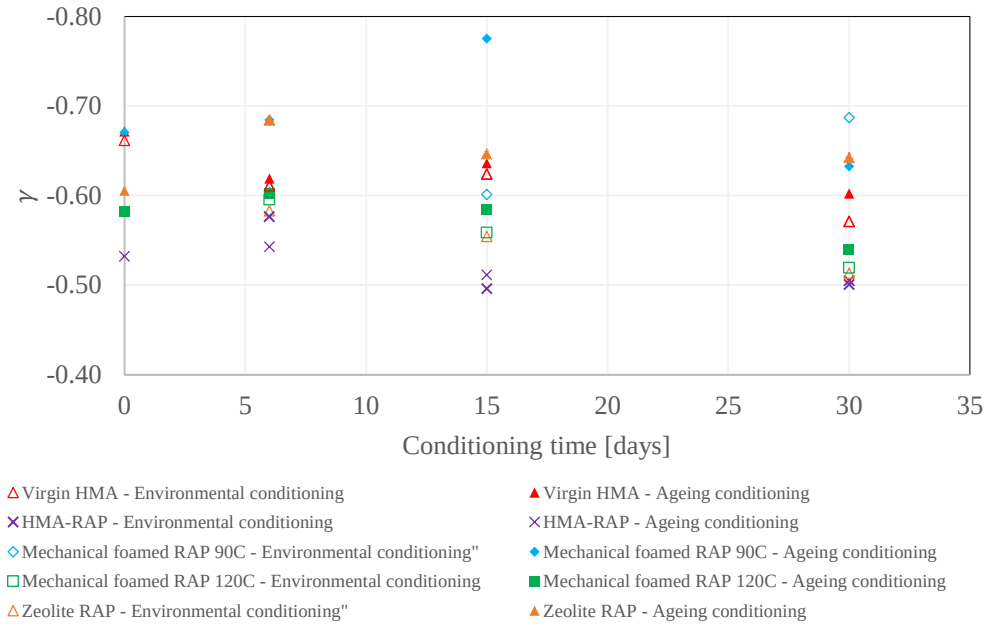


Figure 6.16 γ vs. conditioning time for virgin and FAM mixtures containing RAP and different foaming technologies for both conditioning processes

6.7.2 Black Space mixture parameter

The rheological data obtained from the frequency and temperature sweep testing on the RAP-FBMs was used to calculate the value of the Glover-Rowe parameter at 15ºC and 0.005 rad/ sec by means of equation 21, and evaluate the relative changes in the properties of the mixtures after the environmental conditioning process. Figure 6.17, shows the *G-R* parameter in the black space for the FBM-RAP mixtures, including also the Virgin HMA and the HMA-RAP mixture, as reference mixtures.

In this figure, the Virgin-HMA at the initial condition (i.e. before the environmental conditioning protocol) is at the bottom right, with low G-R and high δ values. As soon as it starts experiencing the environmental conditioning effects the G-R parameter analysis shows that it migrates towards areas where potentially more age hardening occurs (i.e. higher G-R parameter and lower δ values). When RAP material is incorporated, all the FAM mixtures also exhibit higher G-R and lower δ values with respect to the Virgin-HMA at the initial condition, indicating that age hardening is occurring with the RAP material. Furthermore, from this figure, it can also be seen that the Mechanical foamed-RAP mixtures are within the same area of the plot. However after 30 days conditioning, the Mechanical foamed-RAP mixture manufactured at 120ºC shows more ageing hardening compared to the same mixture manufactured at 90ºC, and for the same conditioning period. In general, the Mechanical

foamed-RAP mixtures manufactured at 90ºC and 120ºC exhibit larger differences in the measured G-R and δ values at the different conditioning times compared to the Zeolite-RAP mixtures, as was seen in the corresponding master curves of these materials. The HMA-RAP mixture also shows a characteristic response when subjected to the environmental conditioning. From Figure 6.17, it can be seen that after 6 days conditioning, the rheological changes experienced in this mixture do not differ significantly from its initial condition. However, for further conditioning times, the G-R analysis shows that this mixture migrates towards areas where more age hardening is occurring, exhibiting higher G-R values and significantly lower δ values (decrease from 49º before conditioning to 41º after 30 days conditioning) with respect to its initial condition.

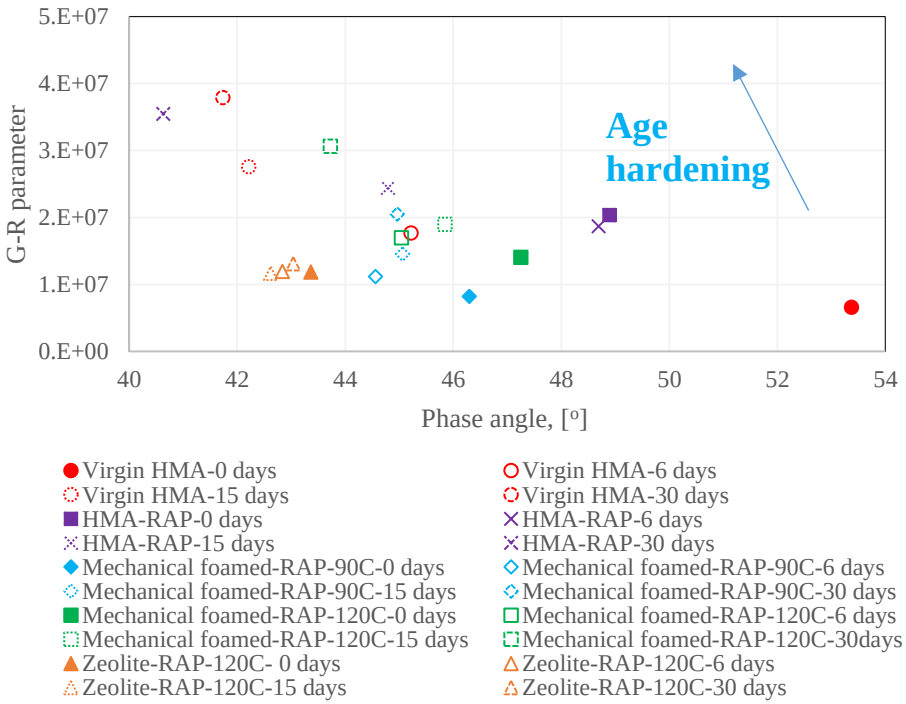


Figure 6.17 Glover-Rowe parameter for virgin and FAM mixtures containing RAP and different foaming technologies at 15ºC and 0.005 rad/sec after environmental conditioning

A similar plot was constructed with the data of the master curves for all the mixtures after ageing conditioning, as shown in Figure 6.18. However, the expected general trends of the mixtures after age conditioning is generally not seen from the G-R analysis. For instance, after age-conditioning, the mixtures show generally higher G-R parameter values with conditioning time, as expected due to the age-hardening related effects, but the δ moves toward higher or lower values with conditioning time. This unusual behaviour of a more viscous response (i.e. higher δ values) after ageing, was also observed in the δ master curves for these mixtures at the low frequencies end, and verified in the δ

isochronal plots at high testing temperatures. Therefore, since the G-R parameter is calculated at a frequency value of 0.0005rad/s (which falls within the low frequencies area of the master δ curves, or high temperatures), the G-R analysis reflects the same response.

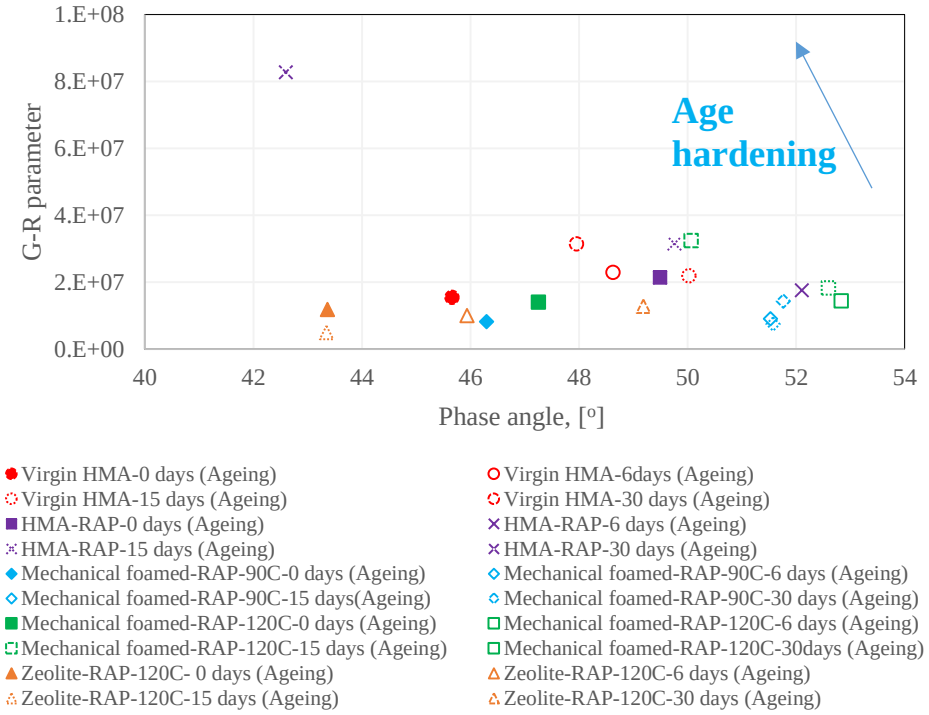


Figure 6.18 Glover-Rowe parameter for virgin and FAM mixtures containing RAP and different foaming technologies at 15ºC and 0.005 rad/sec after pure ageing conditioning

6.8 Conclusions

- Under dry conditions (prior to environmental conditioning), in general, the incorporation of RAP material to all the mixtures resulted in a generalized increase in stiffness compared to that of the Virgin-HMA. Indeed, while complete blending was initially assumed, to target a final penetration of the mixtures containing RAP with the two foaming technologies, equal to that of the Virgin HMA, the degree of blending was found to be a function of the production process and foaming technology used to manufacture the FAM mixtures, as was also seen in Chapter 5.

- In general, the presence of environmental conditions such as moisture and elevated temperatures were observed to modify the rheological properties of the RAP-FBMs. The Virgin-HMA exhibited a pronounced increase in |G*| probably due to oxidative ageing occurring during the

conditioning process in the water bath at 60ºC, generating hardening of the FAM mixtures. The Zeolite-RAP mixture exhibited the highest relative decrease in |G*| among all the mixtures, suggesting that under the presence of moisture, the properties of this mixture could decrease more than in the other materials.

- The changes that occur in the phase angle due to age hardening (more elastic behaviour) for the Virgin HMA and the RAP-FBMs were seen in the low temperature and reduced frequency regions, where the bitumen behaviour is rheologically dominant.

- A comparison of the behaviour of the Virgin HMA and the RAP-FBMs after the environmental conditioning process in the water bath at 60ºC to that of the same mixtures conditioned in an oven at the same temperature but without the presence of water was conducted to factor out the ageing effects observed in the environmental conditioning process. This comparison showed that the ageing occurring in each mixture was different in each type of mixture, and at different combinations of testing temperatures and loading frequencies.

- In general the $|G^*|$ values of all the mixtures increased in both processes. Although the relative ageing occurring in each process was different, the results confirm that there is an effect of ageing occurring in the bitumen present in the FAM materials while present in the water bath at 60ºC. The general increase in the $|G^*|$ of the FAM mixtures after the environmental conditioning could be considered positive in areas where rutting in asphalt mixtures is the main concern in terms of performance. However, in areas where fatigue and low temperature cracking are the main phenomena of interest, additional increase in stiffness of the mixtures could be a major concern.

- Even with the limited conditioning periods, the main result from the $|G^*|$ master curves is that the impact of the environmental conditions on the properties of FBM mixtures with RAP materials is highly dependent on the foamed technology used and on the mixing production conditions (i.e. temperature of the aggregates in the Mechanical foamed RAP mixtures).

Chapter 7 : Combined laboratory ageing and moisture sensitivity assessment of fine RAP-foamed bitumen mixtures using the SATS conditioning protocol

In the previous chapter, the influence of an environmental conditioning process that incorporates the effects of water infiltration and oxidative ageing on the rheological properties of the RAP-FBMs was studied, and a general increase in $|G^*|$ was observed. This chapter evaluates the impact of a more severe combined ageing-moisture sensitivity laboratory conditioning protocol on the rheological properties of the RAP-FBMs by applying, for the first time, the Saturation Ageing Tensile Stiffness (SATS) conditioning on FAM materials. The procedure consists of initial saturation prior to placing compacted cylindrical FAM specimens in a moist, high temperature and pressure environment for 65h. DMA testing was conducted on the FAM specimens before and after SATS conditioning to evaluate the changes in their rheological properties. Furthermore, the properties of the recovered binders of the RAP-FBMs before and after SATS conditioning were evaluated to assess the changes occurring in the bitumen after the SATS conditioning.

7.1 Materials and design of FAM mixtures

For this part of the study, a total of six FAM mixtures were manufactured in the laboratory. These mixtures include a virgin HMA mixture—no RAP material- manufactured at 160°C, with no foaming technology (Virgin-HMA), which corresponds also to the materials used as RAP. A HMA mixture with 50% RAP material manufactured at 160°C, with no foaming technology (HMA-RAP). Furthermore, in order to evaluate the effects of production temperature on the rheological properties of the RAP-FBMs produced through a laboratory foaming plant, three mixtures with a final temperature after mixing of 90, 120, and 160ºC labelled as 'Mechanical foamed-RAP-90°C', 'Mechanical foamed-RAP-120°C', and 'Mechanical foamed-RAP-160°C' respectively were evaluated. For the RAP-FBMs manufactured by the addition of zeolites, the mixture manufactured at 120°C, labelled as 'Zeolite-RAP- 120°C' was incorporated, as it constitutes the main application of this foaming technology (i.e. warm processes). All FAM mixtures were fabricated using the same aggregate composition and bitumen content described in Chapter 3, Section 3.1. The bitumen content for all the FAM mixtures is

10.7% by weight of the total FAM mixture, and they contain fine limestone aggregates (between 0.125 and 1 mm) and limestone filler (less than 65 μ m) in the proportions specified in Section 3.1

The control HMA mixture was fabricated using a 70/100 penetration grade bitumen. In order to characterise this bitumen, penetration at 25°C (BS 2000-49:2007), softening point (BS 2000-58:2007), and Fraass breaking point (BS 2000-80:2007) tests were performed. The same tests were performed on the recovered bitumen from the RAP material. The results from both binders are presented in Table 15.

Table 7-1 Properties of the virgin bitumen and recovered bitumen from RAP

Bitumen	Penetration at 25°C (dmm)	Softening Point (°C)	Fraass breaking point (°C)
Virgin bitumen	66	48	-8
Recovered bitumen	31	59	-8

The RAP material used in this portion of the study was produced in the laboratory as described in Chapter 5, Section 5.1.2. Once the RAP material was produced, the bitumen was recovered by distillation following the BS EN 12697-4:2005, and characterised by performing the same tests as for the virgin bitumen (Table 7-2).

Furthermore, in order to account for the stiffened RAP bitumen present in the mixtures containing 50% RAP, Equations 1 and 2 were used to determine the properties of the virgin bitumen required to obtain a mixture of 70/100 pen (i.e. equal to the virgin bitumen in the HMA mixture for comparison). Table 7-2 shows the properties of each component of the final bitumen blend.

Table 7-2 Bitumen blend design for FAM mixtures with 50% RAP content.

Bitumen	Penetration at 25°C (dmm)	Softening Point (°C)
Virgin bitumen	66	48
Recovered bitumen	31	59
Estimated final bitumen	141	37

Based on these results, a virgin bitumen of 133 dmm penetration at 25°C, softening point of 41°C, and Fraass breaking point of −14°C, was used for the soft virgin bitumen in the HMA-50% RAP mixture and as the foamed bitumen present in the RAP- FBM materials. Based on the previous calculations, it

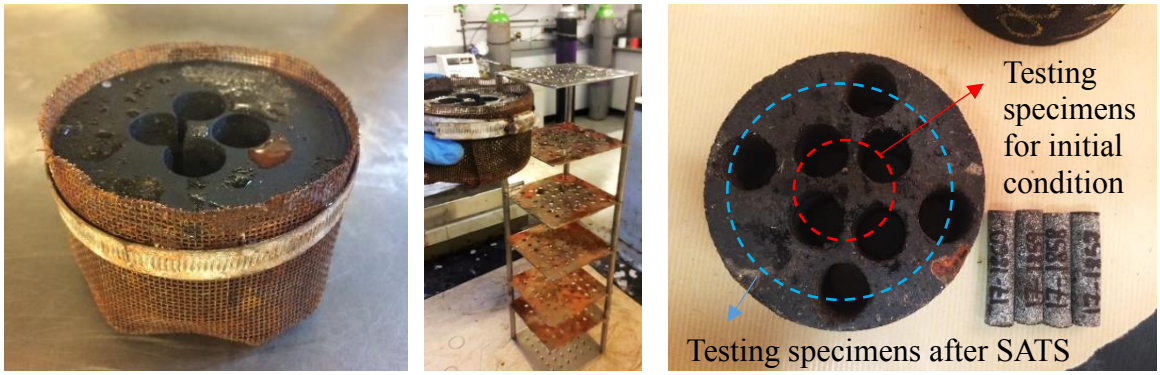
is expected that this virgin bitumen will be blended with the RAP bitumen to produce a final binder with an equivalent 70/100 penetration.

7.2 SATS conditioning method for FAM materials

The SATS procedure was initially developed to test full asphalt mixtures made up of low penetration grade (hard) bitumen (Collop et al., 2004, Airey et al., 2005), and then modified to test asphalt specimens made of higher penetration grade (softer) binders (Grenfell et al., 2012). In the first method, the specimens are subjected to a moisture conditioning process at high temperature (i.e. 85ºC) and high pressure (i.e. 2.1 MPa) for an extended period of time of 65h. In the modified version, the vessel's temperature is kept at 85ºC, and the pressure is reduced to 0.5 MPa for a period of time of 24h. As a first attempt to apply this test on FAM materials, the initial standard test was applied making some modifications on the procedure to test FAM samples in the DMA test. The modified SATS method for FAM mixtures is as follows:

1. Initially, one cylindrical specimen for each FAM mixture with dimensions of 100mm in diameter by 60 mm in height is compacted in the laboratory using the Superpave gyratory compactor (SGC) following the BS EN 12697-31:2007. For this study, the specimens were compacted targeting a density of 2056 kg/m^3 (i.e. a target air void content of the compacted FAM specimens of 10%).
2. From the 100mm diameter SGC FAM specimen, the top and bottom parts are trimmed to obtain a specimen of 50mm in height, and four cylindrical samples of 50 mm in height by 12.5 mm in diameter are extracted from the centre of the SGC core using a coring barrel. These samples are retained for DMA testing for the "initial condition".
3. The dry mass of the cored SGC specimen is determined.
4. The specimen is then immersed in distilled water at 20ºC and vacuum saturated using a residual pressure of (400-700 mbar) 65 kPa for 30 min.
5. The wet mass of the specimen is then determined by weighing, and the percentage saturation is calculated, referred to as 'initial saturation'. The specimen is then embedded in a metal mesh to avoid deformation during conditioning (Figure 7.1 a).
6. The SATS pressure vessel is partially filled with distilled water until the level is between the fourth and fifth position (P4 and P5 in Figure 2.24). The pressure vessel and the water are maintained at the target temperature of 85ºC for at least 2h prior to introducing the specimens.

7. The saturated asphalt specimen is then placed into the pressure vessel, in the top position of the specimen tray (P1 in Figure 7.1 b). The standard SATS test holds five cores at a time, which are positioned along the specimen tray (P1 to P5, as explained in the literature review in Chapter 2). For the purpose of this study, and since only one SGC specimen is manufactured per FAM mixture, the top position was selected, as it is the most critical in terms of moisture conditioning. The vessel is then sealed and the air pressure is gradually raised to 2.1 MPa.
8. The specimen is maintained at these testing conditions (i.e. 2.1MPa and 85ºC) for 65 hours.
9. After 65h, the target vessel temperature is reduced to 30ºC and the vessel is left for 24h to cool. When the pressure vessel's display temperature has reduced to 30ºC (after the 24h cooling period), the air pressure is gradually released. When the vessel has achieved atmospheric pressure, it is opened and the specimen is removed. The specimen is then surface dried and weighed in air. The percentage saturation calculated at this stage is referred to as the 'retained saturation'.
10. Four cylindrical samples of 50 mm in height by 12.5 mm in diameter are extracted from the outer part of the specimen avoiding contact with the already cored holes, using a coring barrel (Figure 7.1). These samples are retained for DMA testing for the "final condition".



a) b) c)

Figure 7.1 SATS test method for FAM material. a) SGC - FAM specimen embedded in a metal mesh, b) specimen placed in the top position of the specimen tray, and c) cored FAM samples for testing under initial condition and after SATS conditioning.

The viscoelastic material properties (i.e. $|G^*|$ and δ) of the FAM samples before and after SATS conditioning, were determined by means of DMA testing, using the testing conditions described in Section 3.2.1.

The air void content for the cylindrical FAM testing specimens were determined by measuring their volumetric properties, and are presented in Figure 7.2. The specimens obtained from the inner concentric zone of the SGC core (Zone A in Figure 7.2), were used for DMA testing at the initial condition (before SATS conditioning). The specimens obtained from the outer zone of the SGC core (Zone B in Figure 7.2), on the contrary, were retained for testing after SATS conditioning. The air void content values in this figure correspond to the average of 4 specimens obtained from each zone.

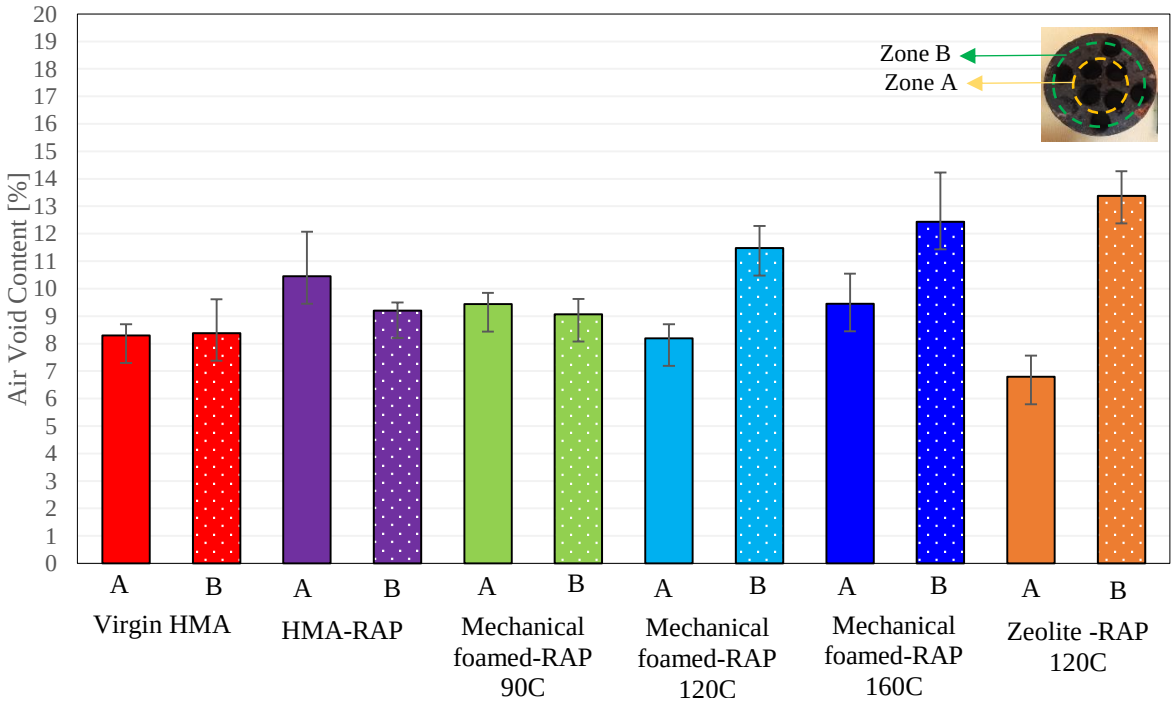


Figure 7.2 Air void content of FAM specimens for DMA testing for all the mixtures

7.3 Results of the effects of SATS conditioning on the rheological properties of the RAP-FBMs

7.3.1 Initial condition of the RAP-FBMs

The average dynamic shear modulus, $|G^*|$ master curves at a reference temperature of 25°C for all the FAM mixtures prior to SATS conditioning are presented in Figure 7.3.

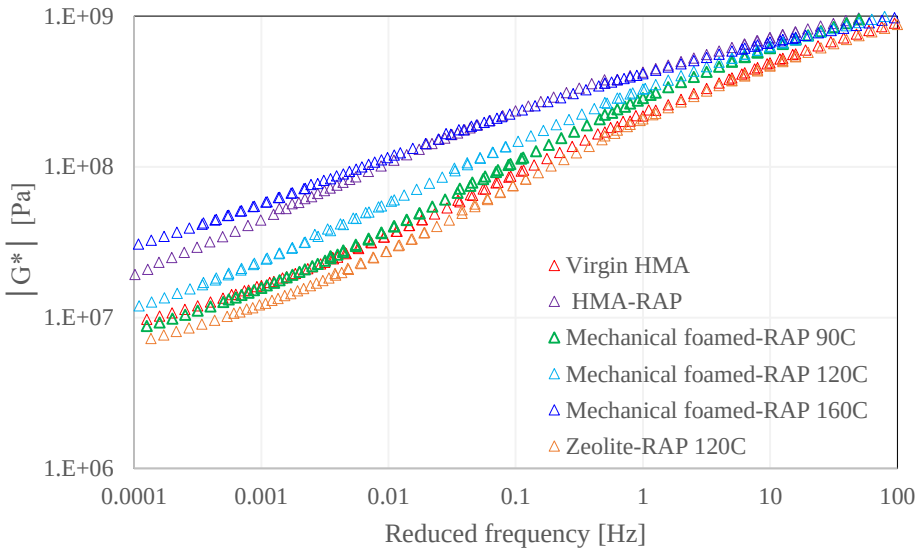


Figure 7.3 Master curves of |*G| for all mixtures at the initial condition.**

Under dry conditions, Figure 7.3 shows once more that the FAM mixtures containing 50% RAP material and produced at different temperatures (90-160ºC) exhibit a different shift of the $|G^*|$ master curves with reference to the Virgin HMA, as was observed and discussed in Chapters 5 and 6. Figure 7.3 also confirms that full blending between the hard bitumen from RAP and the soft virgin bitumen in the HMA-RAP mixture (no foaming technology) did not occur as the resultant mixture exhibited a different final penetration than the one expected (70/100 pen, as the Virgin HMA). This behaviour (incomplete blending) occurred in the Mechanical foamed-RAP mixture manufactured at 160ºC as well, exhibiting consistently the highest $|G^*|$ values among all the mixtures. This behaviour breaks the analogy of complete blending as was also discussed in Chapter 4, however these results suggest that the dual effects of high production temperature of 160ºC and the incorporation of 50% RAP in the FAM materials, are critical in the stiffening effect of the materials. At production temperatures of or below 120ºC, the FBM mixtures containing RAP exhibited a softening effect with respect to the HMA-RAP, as depicted by lower |G*| values. These results suggest that the level of blending between old-new materials occurred to a different extent in each mixture, depending on the final mixing temperature and production process.

The black space plot in Figure 7.4, shows that the Mechanical foamed-RAP produced at 160ºC exhibits the lowest δ across the $|G^*|$ values, indicating an increase in elastic behaviour for these materials. The Black diagrams curves for the Mechanical foamed-RAP mixtures produced at 90 and 120ºC, and the Zeolite-RAP mixture are similar to that of the Virgin HMA, indicating that the

rheological properties of these mixtures are comparable to those of the Virgin HMA. The HMA-RAP material exhibits a more elastic response (lower δ values) with respect to the Virgin HMA, which are more significant at the high-$|G^*|$ end. This increased elastic response is a result of the hard bitumen from RAP, which results in an increased stiffness and more elastic response.

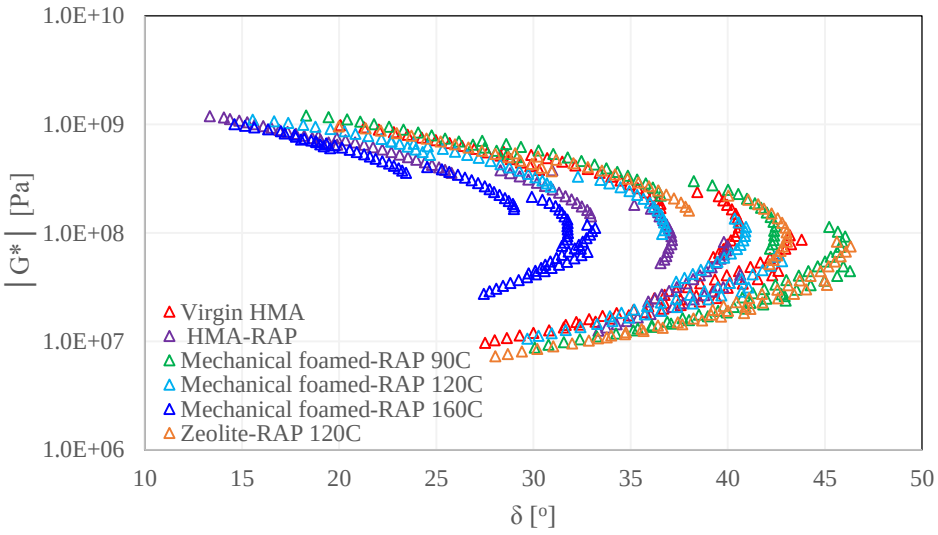


Figure 7.4 Black diagrams for all the mixtures in dry condition.

A statistical analysis was also conducted on the raw data as in Chapters 5 and 6, using independent sample t-tests with a confidence interval of 95% to determine the significance of the production temperature, the foaming technology, and the loading frequency on the $|G^*|$

The Mechanical foamed-RAP mixture manufactured at 90ºC shows no statistical difference compared to the Virgin HMA at testing temperatures of or above 35ºC (low-reduced frequencies in the master curves). At temperatures below 35ºC (high-reduced frequencies in the master curves), these mixtures are statistically different. The Mechanical foamed-RAP mixture manufactured at 120ºC is not statistical different from the Virgin HMA at testing temperatures of 15 and 65ºC, which corresponds to the high and low reduced frequency ends in the master curves. At testing temperatures between 15 and 65ºC, both mixtures are statistically significant. Furthermore, the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC do not exhibit statistical significance between each other at all frequencies. The Mechanical foamed-RAP manufactured at 160ºC and the HMA-RAP mixtures are statistically different from the Virgin HMA at all reduced frequencies. Furthermore, the Zeolite-RAP mixture shows statistical significance from the Virgin HMA at low reduced frequencies (high testing temperatures). At higher reduced frequencies (low testing temperatures), the mixtures are statistically different.

The statistical analysis confirms that complete blending between the RAP and virgin binder did not occur in the control HMA-RAP mixture, as this mixture presents statistical difference from the Virgin HMA. Similar results were obtained with the Mechanical foamed-RAP mixture manufactured at 160ºC, thus confirming the behaviour observed in the master curves in Figure 7.3, and validating the results obtained in Chapter 6. These results imply that a different mechanism occurs when 50% RAP material and a mixing temperature of 160ºC in FAM materials are employed, as was also explained in Chapter 5, generating a significant increase in the stiffness and elastic behaviour of the materials. The Mechanical foamed-RAP mixture manufactured at 120ºC, exhibits statistical difference from the Virgin HMA, whereas the Mechanical foamed-RAP mixtures manufactured at 90ºC and the Zeolite-RAP mixture manufactured at 120ºC exhibits little statistical difference from the Virgin HMA. These results indicate that when foaming technologies are incorporated and at lower mixing temperatures of 90 and 120ºC, the stiffening effect of the mixtures is reduced, thus generating mixtures with comparable properties to those of the Virgin HMA.

7.3.2 Saturation of the RAP-FBMs

Table 7-3 shows the saturation level of the cores that were subjected to SATS conditioning. It is important to mention that these saturation levels correspond to the cores of the SGC FAM specimens and not the saturation levels in the FAM specimens that were tested in the DMA.

Table 7-3 Saturation levels of the SGC FAM specimens for all the mixtures

Mixture	Initial saturation (%)	Final saturation (%)
Virgin HMA	38.5	36.4
HMA-RAP	53.5	43.6
FBM-RAP 90C	30.4	28.2
FBM-RAP 120C	45.6	43.1
FBM-RAP 160C	38.2	36.0
Zeolite-RAP 160C	33.8	31.3

7.3.3 Effects of SATS conditioning on the rheological properties of the RAP-FBM mixtures

The master curves of $|G^*|$ for all the mixtures at the initial condition and after SATS conditioning, are presented in Figure 7.5. The $|G^*|$ master curves in this figure show that the Virgin HMA, the

Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC, and the Zeolite-RAP mixture manufactured at 120ºC exhibit a clear shift towards higher |*G**| values after SATS conditioning, becoming more relevant at lower reduced frequencies. The Mechanical foamed-RAP mixture manufactured at 160ºC and the HMA-RAP mixture instead, show a pronounced flattening effect of the master curves after SATS conditioning, which results in a shift towards higher $|G^*|$ values at low reduced frequencies, and lower $|G^*|$ at high reduced frequencies, after SATS conditioning.

The increase in |*G**| values after SATS conditioning, implies that the mixtures exhibited a stiffening effect with respect to its initial condition. This stiffening effect of the FAM mixtures, could be explained after considering the mechanisms by which the combined effect of ageing and moisture during SATS conditioning could act within the FAM mixtures. When the FAM materials are subjected to the wet environment, the first mechanism by which moisture can reach their internal microstructure is by water infiltration, which will depend mainly on the air voids. Recalling that these materials are composed of a combination of a rich bitumen phase (i.e. around 10.7%), fine particles (i.e. below 1 mm), and air void content (which is around 10% for the considered mix design), these air voids are smaller in size compared to those of conventional HMA materials (Vasconcellos, 2010). Therefore, the small size air voids along with the high bitumen phase, could impact the rate at which moisture can reach the inner portion of the specimen and allow damage to occur, whereas the elevated temperatures would certainly lead to some ageing and stiffening of the materials, which explains the increase in $|G^*|$ in the master curves. These results are in agreement with those presented in Chapter 6 where the FAM materials were subjected to an environmental conditioning that included the presence of moisture and oxidative ageing, and the $|G^*|$ exhibited a general increase up to 30 days conditioning time. Thus, suggesting that in the presence of moisture and high temperature environments the ageing-related hardening effects dominate the response of the FAM materials, over the degradation effects that can be generated by the presence of moisture. This point is also supported by the fact that after SATS conditioning, the saturation levels of the SGC specimens were between 36 and 45% (Table 7-3), which are significantly lower compared to those reported by other researchers for full asphalt mixtures on specimens located at the same position (P1), and using the same conditioning protocol, which are around 80% (Airey et al, 2005), indicating a partial saturation of the FAM specimens. In addition, these results imply that in FAM materials the time needed for water to penetrate their internal structure and degrade their mechanical capacity is longer, as was also observed in Chapter 6, where even after 30 days conditioning in a water bath the $|G^*|$ of the mixtures exhibited a general increase with respect to the initial $|G^*|$ values of the evaluated mixtures.

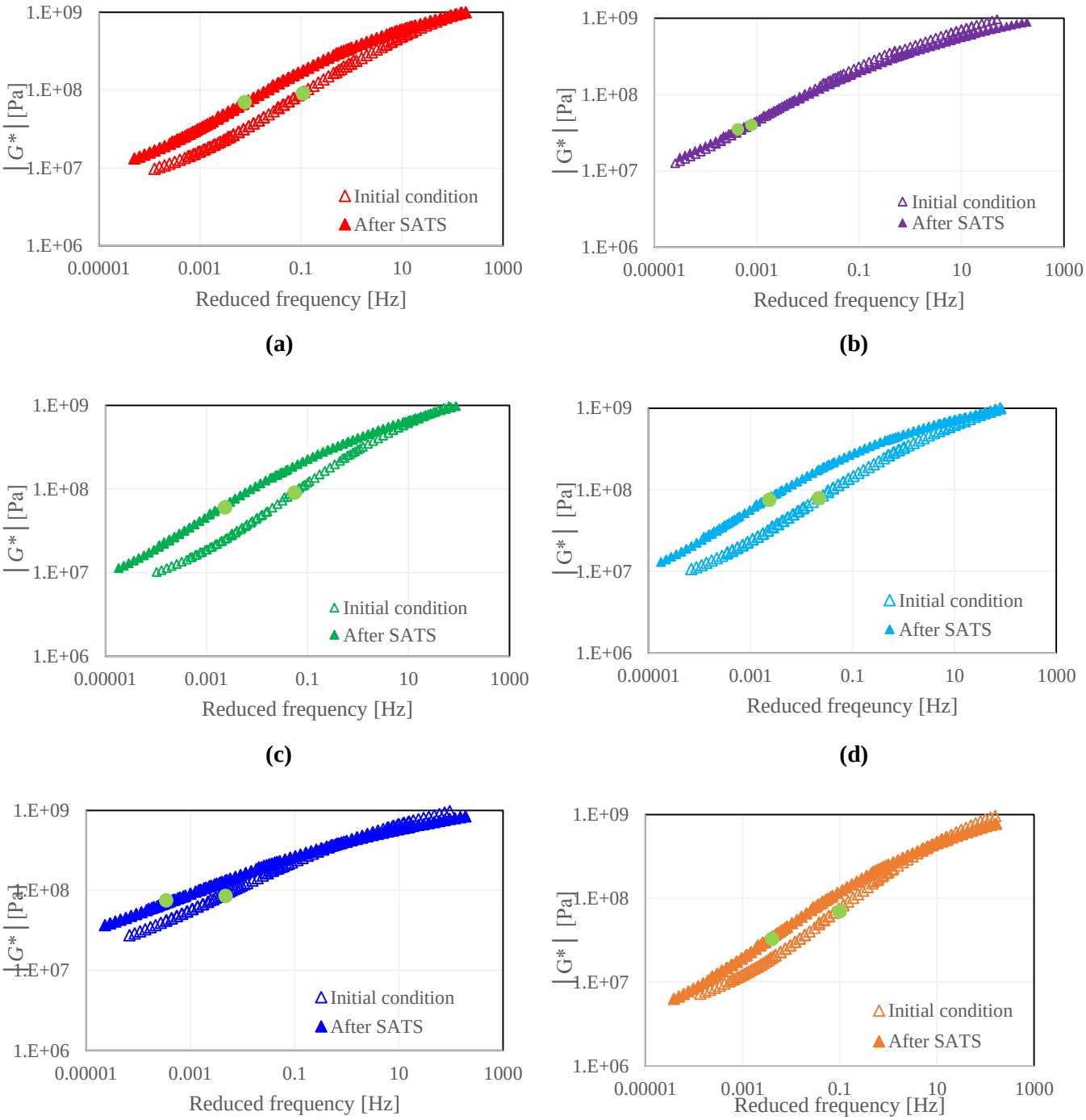
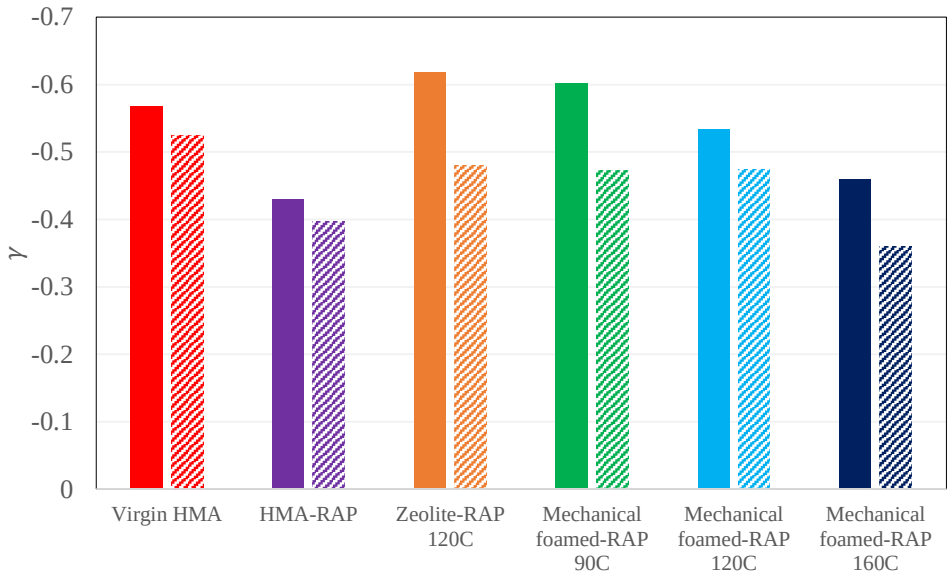
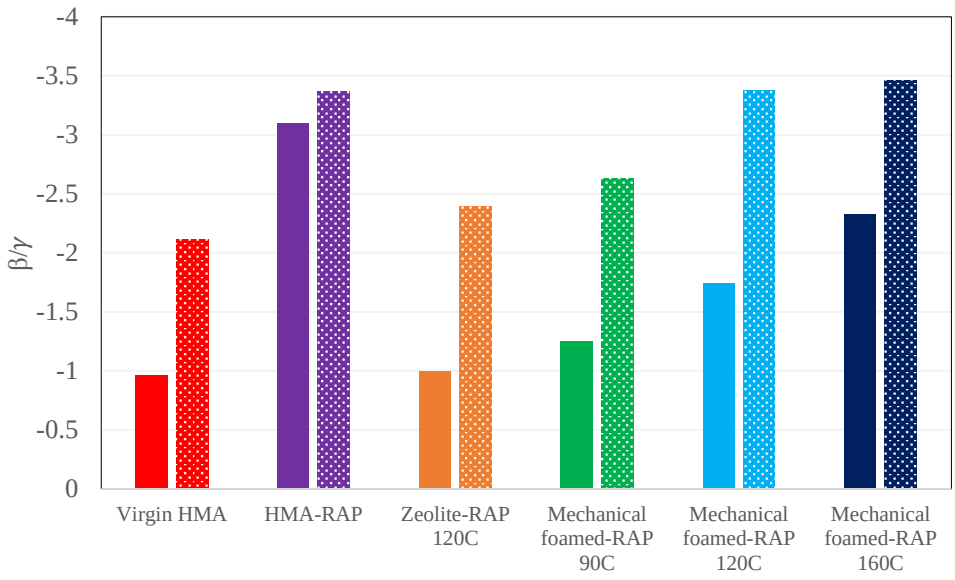


Figure 7.5 $|G^*|$ Master curve for: (a) Virgin HMA, (b) HMA-RAP, (c) Mechanical foamed-RAP 90C, (d) Mechanical foamed-RAP 120C, (e) Mechanical foamed-RAP 160C and (f) Zeolite-RAP 120C

In recent studies to assess the oxidative ageing that occurs within an asphalt mixture, the physical significance of the fitting parameters (β and γ) that are related to the $|G^*|$ master curve shape in the standard sigmoidal function has been used (Mensching et al. 2015), as described in the literature review in Chapter 2. Therefore, in order to further evaluate the ageing-related hardening effects of SATS conditioning observed in the $|G^*|$ master curves of the RAP-FBMs, the average data of the $|G^*|$ for all the mixtures were fitted using the standard sigmoidal function by means of equation 20, and the parameters β and γ were used to quantify the changes in the shape of the $|G^*|$ master curves after SATS conditioning, as displayed in Figure 7.6. The differences in both parameters after SATS conditioning are also presented in Table 7-4. On one hand, γ parameter describes the slope of the $|G^*|$ master curve, meaning that higher values of this parameter (lower absolute values) are related to more age hardening in the mixtures due to the flattening effect of the master curve. Indeed, after SATS conditioning all materials exhibit higher γ values (lower absolute values) (Figure 7.6a). On the other hand, β parameter controls the inflection point position of the flattening effect of the master curve after ageing. The inflection point (inflection frequency in the $|G^*|$ master curve), which is calculated as $10^{-\beta/\gamma}$ moves to the left towards lower frequencies, with increase in RAP or ageing of asphalt mixtures (Mensching et al. 2015). In this sense, after SATS conditioning the inflection point for each mixture moves to the left, as depicted by the green dots in the $|G^*|$ master curves in Figure 7.5, or lower β/γ values (higher absolute values) in Figure 7.6b.



a)



b)

Figure 7.6 Plots of (a) shape parameter γ, and (b) log of inflection frequency

Table 7-4 Differences in γ and $-\beta/\gamma$ values after SATS conditioning

Mixture type	γ		Difference*	$-\beta/\gamma$		Difference
	Before SATS	After SATS		Before SATS	After SATS	
Virgin HMA	-0.57	-0.52	7.6	-0.96	-2.11	119.5
Mechanical foamed-RAP 90C	-0.60	-0.47	21.5	-1.25	-2.63	109.7
Mechanical foamed-RAP 120C	-0.53	-0.47	11.0	-1.74	-3.38	93.8
Mechanical foamed-RAP 160C	-0.46	-0.36	21.5	-2.32	-3.46	49.2
Zeolite-RAP 120C	-0.62	-0.48	22.5	-1.00	-2.39	140.0
HMA-RAP	-0.43	-0.40	7.5	-3.10	-3.37	8.6

Based on data shown in Figure 7.6 and Table 7-4, inferences can be drawn on the contributions of these parameters and differentiate the ageing effects in each type of mixture. For instance, looking at the Virgin HMA as the reference mixture, this mixture exhibits high γ values and also greater differences in the inflection point frequency (related to $-\beta/\gamma$) after SATS test. This indicates an overall increase in the stiffness and age-hardening effects in this mixture. Similarly, the Mechanical foamed-

RAP mixtures manufactured at 90 and 120ºC, and the Zeolite-RAP mixture manufactured at 120ºC exhibit close γ values to those of the Virgin HMA and also great differences in the inflection point frequency, indicating that that these mixtures exhibit high ageing. It is interesting to notice from Figure 7.6 and data provided in Table 7-4 that the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC, and the Zeolite-RAP manufactured at 120ºC, exhibit similar γ values after SATS conditioning and the changes in the inflection frequency after SATS conditioning are lower for higher mixing temperatures, thus indicating lower age-hardening effects with higher production temperature of the mixtures. The Mechanical foamed-RAP mixture manufactured at 160ºC and the HMA-RAP, on the contrary, exhibit the lowest γ values indicating lower slopes with respect to the rest of the mixtures, and also the lowest changes in the inflection point frequency after SATS conditioning, thus indicating less age-hardening effects after ageing. These results corroborate the observations made from the $|G^*|$ master curves and imply that the age-hardening effects are different with the initial stiffness of the mixtures, where the stiffest mixtures exhibited less ageing effects.

The complete rheological behaviour ($|G^*|$ and δ) for all the mixtures, was plotted in the black space in Figure 7.7. In these plots, the results at low temperatures are in the top part of the plot and the results at high temperatures are in the lower part of the plot. After SATS conditioning, the mixtures reveal high elastic response (lower δ values) at low temperatures, due to the age-hardening of the bitumen. After the inflection point, at high temperatures, the curves for both conditions (before and after SATS conditioning) overlap for the Virgin HMA, the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC, and the Zeolite-RAP mixture manufactured at 120ºC. The Mechanical foamed-RAP mixture manufactured at 160ºC and the HMA-RAP mixture, exhibit a pronounced elastic behaviour after SATS conditioning, as displayed by lower phase angle values for the wide range of $|G^*|$. Indeed these mixtures exhibited the highest $|G^*|$ and lowest δ values after SATS conditioning, indicating an overall increase in the hardness and brittleness of these mixtures. Thus, these results corroborate that the incorporation of 50% RAP material at high mixing temperatures of 160ºC is critical in the response of the mixtures for fatigue and thermal cracking resistance.

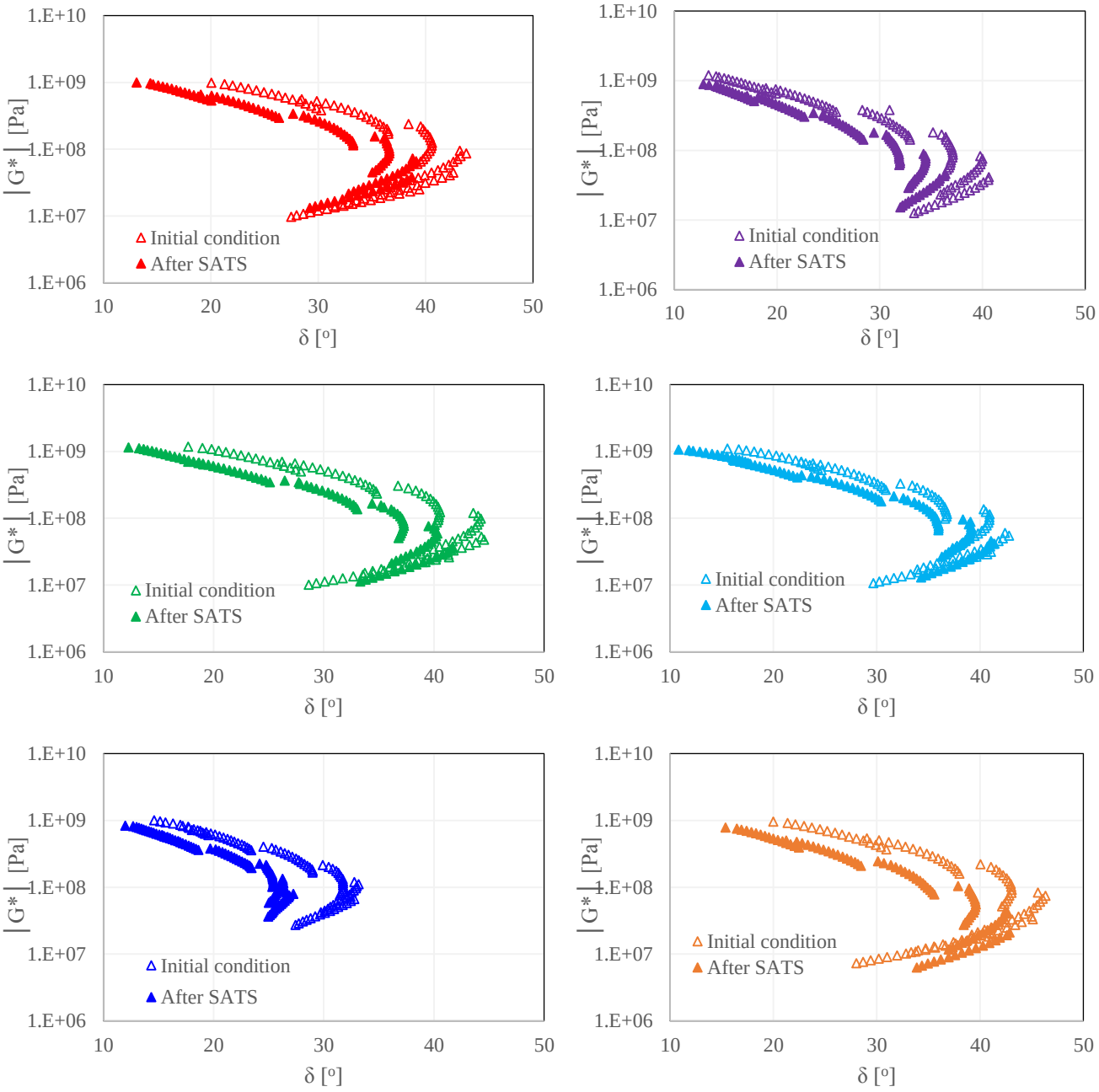


Figure 7.7 Black diagrams for: (a) Virgin HMA, (b) HMA-RAP, (c) Mechanical foamed-RAP 90C, (d) Mechanical foamed-RAP 120C, (e) Mechanical foamed-RAP 160C and (f) Zeolite-RAP 120C

With the objective of better quantifying the effect of SATS conditioning on the $|G^*|$ of the mixtures, the average $|G^*|$ values at all temperatures and two constant frequencies of 3.162Hz and 10Hz for each mixture, was used to calculate the ratio of the $|G^*|$ values after SATS conditioning with respect to the initial condition, as displayed in Figure 7.8.

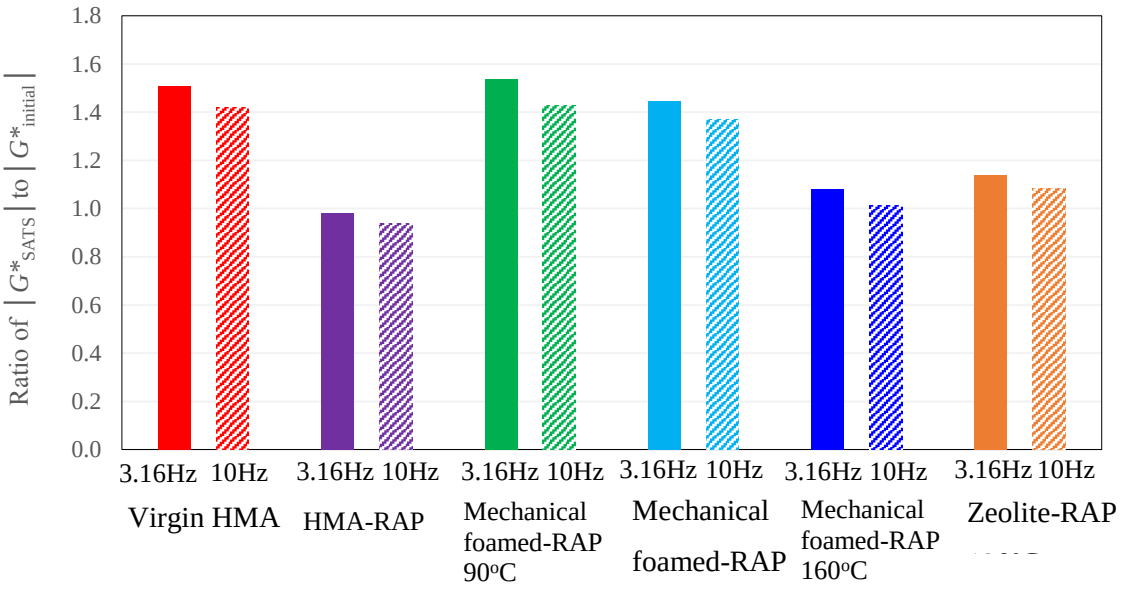


Figure 7.8 Dynamic Shear modulus ratios at 3.16Hz and 10 Hz for all the mixtures

Figure 7.8 shows that the Virgin HMA exhibits high ratios. This high stiffening could be due to the softer consistency of the bitumen present in this mixture compared to the RAP mixtures, which contains an already aged RAP bitumen. Furthermore, in terms of the RAP mixtures and looking at the Virgin HMA as the control reference mixture, it can be observed that the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC exhibit similar ratios, while the Mechanical foamed mixtures manufactured at 160ºC and HMA-RAP mixture exhibit the lowest ratios. The Zeolite-RAP mixture exhibit lower ratios than the Mechanical foamed-RAP mixtures manufactured at low temperatures of 90 and 120ºC, and also to those of the Virgin HMA. These results imply that the age-hardening related effects after SATS conditioning for the mixtures containing RAP, are different in each type of mixture, as was discussed previously, where the mixtures with high initial stiffness exhibited less ageing than those of a lower initial stiffness. Indeed, the Mechanical foamed mixtures manufactured at 160ºC and HMA-RAP mixture exhibit ratios closer to 1, thus corroborating that these mixtures did not experience significant age hardening. These differences could be attributed to the degree of blending or interaction between the old-new binders occurring in each mixture, as was already observed from the master curves for the initial condition of the mixtures. In addition, the differences in the air void content could also be contributing to some of the stiffening observed. The differences in the $|G^*|$ ratios, are studied in more detail in Figure 7.9, where the ratios were plotted as a function of the initial stiffness of each material.

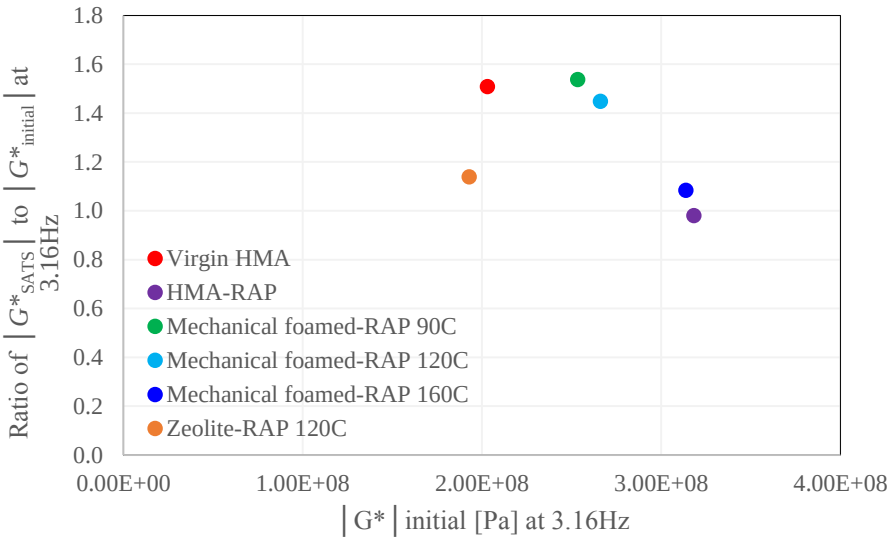


Figure 7.9 Dynamic Sher modulus ratios vs. initial stiffness of the mixtures

Figure 7.9 corroborates that the mixtures with higher initial stiffness (HMA-RAP and Mechanical foamed-RAP 160ºC), have the lowest $|G^*|$ ratios, meaning that the stiffer mixtures experienced less age-hardening effects after SATS condition, as was also observed from the $|G^*|$ and master curves and the analysis from the shape parameters. Figure 7.9 also shows that the Mechanical foamed-RAP mixtures manufactured at 90 and 120ºC, exhibit similar ratios to those of the Virgin HMA, which could be related to the similarity in the properties of these mixtures as was observed in the master curves in Figure 7.5. The general tendency is not seen in the Zeolite-RAP mixture. This mixture exhibits lower ratios, than those reported for the Mechanical foamed-RAP mixtures and the Virgin HMA, having similar initial $|G^*|$ values. However, this result reflects that the effects of ageing were different with type of mixture as well.

7.4 Results of the effects of SATS conditioning on the rheological properties of the recovered binders after SATS tests

To study the effects of SATS conditioning on the rheological properties of the bitumen present in the RAP-FBMs, samples from extracted binders before and after SATS conditioning were evaluated by means of Dynamic Shear Rheometer (DSR). Binders were recovered by distillation following the BS

EN 12697-4:2005. Frequency and temperature sweep tests were performed, employing the testing conditions described in Section 3.2.2.

7.4.1 Initial condition of the binders

Initially, the master curves of |*G**| at a reference temperature of 25ºC for the recovered binders of each mixture at the initial condition (i.e. prior to conditioning in the SATS protocol) are reported in Figure 7.10.

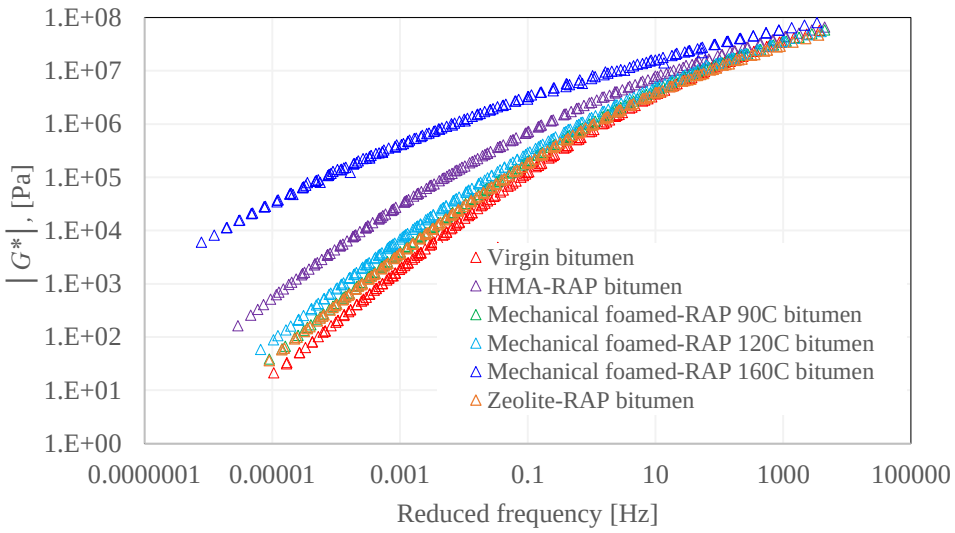


Figure 7.10Master curves of G* for all recovered binders at the initial condition

This figure reveals that recovered binders from the mixtures containing RAP material exhibited an increase in |*G**| values, as depicted by a vertical shift of |G*| curves with reference to the Virgin bitumen, being more significant for binders recovered from the Mechanical foamed-RAP mixture manufactured at 160ºC, and the HMA-RAP mixture manufactured also at 160ºC. Comparing Figure 7.1 with Figure 7.3 (equivalent $|G^*|$ master curves for the corresponding FAM mixtures), some key trends can be noticed. For instance, results in this figure show that the recovered binder from the HMA-RAP mixture exhibit different properties to that of the virgin bitumen, thus indicating that the blend between the hard (RAP) binder and the soft virgin binder did not result in a blend with similar penetration grade to that of the bitumen present in the Virgin HMA, as was theoretically assumed. These findings reflect the contribution of other factors in the mixtures, which are influencing the characteristics of the produced mixtures. A similar behaviour is observed from the bitumen recovered from the Mechanical foamed-RAP mixture manufactured at 160ºC in Figure 7.10, which exhibits consistently the highest $|G^*|$ values across the reduced frequency axis among all recovered binders, explaining the increased stiffness observed in the corresponding FAM mixture. However, the

properties ($|G^*|$ values) of the Mechanical foamed-RAP mixture in Figure 7.3 exhibit similar values to those of the HMA-RAP mixture, and the same behaviour is not reflected in the corresponding binders $|G^*|$ master curves in Figure 7.1, where the properties of these two binders exhibit different $|G^*|$ values. The increased stiffness observed in the HMA-RAP mixture in Figure 7.3 could be partly attributed to the aggregate skeleton within the FAM mixtures, even if there is a rich-bitumen phase in these materials, the properties of the aggregate skeleton are still able to control the response of the mixture.

Furthermore, another key trend to notice in Figure 7.10, is the increase in stiffness of the binders recovered from the Mechanical foamed-RAP mixtures, with production temperature. The increase in stiffness across the reduced frequency axis is consistent, with the stiffening at a production temperature of 90^{o}C being of a similar magnitude as the stiffening of the recovered binder from the Zeolite-RAP mixture manufactured at 120^{o}C. Similar trends for the Mechanical foamed-RAP mixtures with production temperature are seen in Figure 7.1. These results corroborate that the difference in the degree of hardening observed in the RAP-FBMs, are related to the different levels of interaction between the RAP bitumen and the virgin bitumen present in each mixture, being different with production temperature of the mixtures. Short-term ageing with production temperature may be also contributing to these effects.

In terms of the Zeolite-RAP mixture, this mixture exhibits a slight decrease in the $|G^*|$ values with reference to the Virgin HMA in Figure 7.1, while the corresponding binder in Figure 7.3 shows a slight increase with reference to the Virgin binder. The heterogeneity present in the FAM materials could be contributing to this difference between the FAM and recovered binders properties. On the other hand, during the extraction process the binders are forced to mix, making them different than the material tested as part of the mixture itself. Hence, leading to different correlations between these materials.

7.4.2 Effects of SATS conditioning on the rheological properties of the binders present in the RAP-FBM mixtures

The master curves, isochronal plots, and Black diagrams for the recovered binders from each mixture are all similar and, therefore the results will only be presented for one bitumen with the understanding that the commentary applies to all 6 recovered binders.

The master curves of $|G^*|$ and δ for the bitumen recovered from the Zeolite-RAP mixture in Figure 7.11, show a regular increase in $|G^*|$ and decrease in δ after SATS conditioning, corresponding to an increased hardness and increase in elastic behaviour of the bitumen due to oxidative ageing.

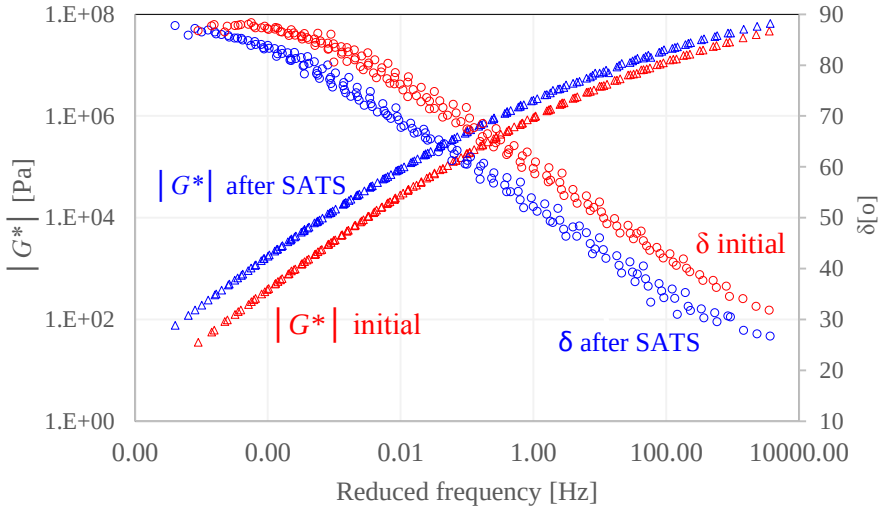


Figure 7.11 G* and δ master curve for Zeolite-RAP 120°C

The smooth shifting of the $|G^*|$ and δ master curves in Figure 7.11, indicates that the TTSP is applicable for the recovered binders and that there is an equivalency between time (loading frequencies) and temperature. This fact allows a limited combination of temperatures and frequencies to get a general overview of the changes in rheological characteristics of the binders for further evaluation. In this sense, the $|G^*|$ and δ values at one temperature of 25°C and two loading frequencies of 0.16Hz and 1.6Hz for all the recovered binders are presented in Table 7-5.

Table 7-5 Changes in $|G^*|$ and δ after SATS conditioning

Bitumen	Property	Unaged	SATS conditioning	Ageing index (SATS condition/Unaged/)
Virgin bitumen	$G^*_{25C,\ 0.152Hz}$ [Pa]	178100	1504000	8.44
	$G^*_{25C,\ 1.6Hz}$ [Pa]	1033000	5764000	5.58
	$\delta_{25C,\ 0.152Hz}$ [°]	72	57	-
	$\delta_{25C,\ 1.6Hz}$[°]	65	50	-

Bitumen	Property	Unaged	SATS conditioning	Ageing index (SATS condition/Unaged/)
HMA-RAP bitumen	$G^*_{25C,\ 0.152Hz}$ [Pa]	921900	2220000	2.41
	$G^*_{25C,\ 1.6Hz}$ [Pa]	3216000	5884000	1.83
	$\delta_{25C,\ 0.152Hz}$ [^{o}]	52	52	-
	$\delta_{25C,\ 1.6Hz}$[^{o}]	47	40	-
Mechanical foamed-RAP 90^{o}C bitumen	$G^*_{25C,\ 0.152Hz}$ [Pa]	282100	887100	3.14
	$G^*_{25C,\ 1.6Hz}$ [Pa]	1401000	3315000	2.37
	$\delta_{25C,\ 0.152Hz}$ [^{o}]	66	56	-
	$\delta_{25C,\ 1.6Hz}$[^{o}]	59	49	-
Mechanical foamed-RAP 120^{o}C bitumen	$G^*_{25C,\ 0.152Hz}$ [Pa]	391600	956700	2.44
	$G^*_{25C,\ 1.6Hz}$ [Pa]	1733000	3443000	1.99
	$\delta_{25C,\ 0.152Hz}$ [^{o}]	61	54	-
	$\delta_{25C,\ 1.6Hz}$[^{o}]	55	47	-
Mechanical foamed-RAP 160^{o}C bitumen	$G^*_{25C,\ 0.152Hz}$ [Pa]	3920000	7979000	2.04
	$G^*_{25C,\ 1.6Hz}$ [Pa]	8360000	16820000	2.01
	$\delta_{25C,\ 0.152Hz}$ [^{o}]	45	31	-
	$\delta_{25C,\ 1.6Hz}$[^{o}]	33	28	-
Zeolite-RAP 120^{o}C bitumen	$G^*_{25C,\ 0.152Hz}$ [Pa]	259000	676700	2.61
	$G^*_{25C,\ 1.6Hz}$ [Pa]	1262000	2657000	2.11
	$\delta_{25C,\ 0.152Hz}$ [^{o}]	65	58	-
	$\delta_{25C,\ 1.6Hz}$[^{o}]	58	51	-

The $|G^*|$ and δ data in Table 7-5 provides the same findings, seen in Figure 7.11, of an increase in stiffness and elastic behaviour after SATS conditioning. Furthermore, in terms of the ageing indices for $|G^*|$, and using the bitumen recovered from the Virgin HMA mixture as the reference control binder, results show that the recovered binders from the RAP mixtures exhibit lower ageing indices, indicating that the effects of oxidative ageing after SATS conditioning are less severe for the stiffer binders. Figure 7.12 shows this relationship, by plotting the initial stiffness (unaged) of the binders at 0.152Hz and 25^{o}C against the ageing index. The same plot was constructed with the data at 1.6Hz, obtaining the same results, thus was not presented here for conciseness.

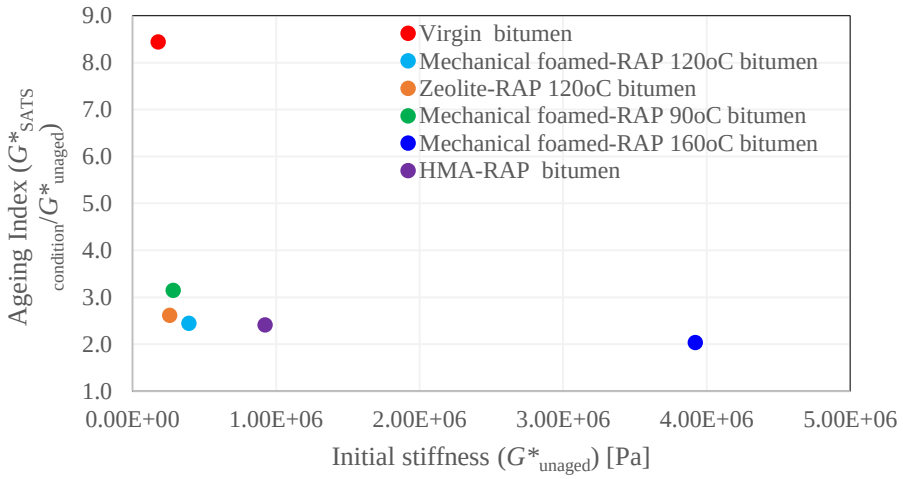


Figure 7.12 Initial stiffness vs. Ageing Index

The black diagram curves for the binders recovered from the Mechanical foamed-RAP mixture manufactured at 160ºC before and after SATS conditioning in Figure 7.13 show a continuous shift of the curves towards lower δ values after SATS conditioning. This shift is caused by the dual actions of increase in $|G^*|$ and decrease in δ as seen in the master curves.

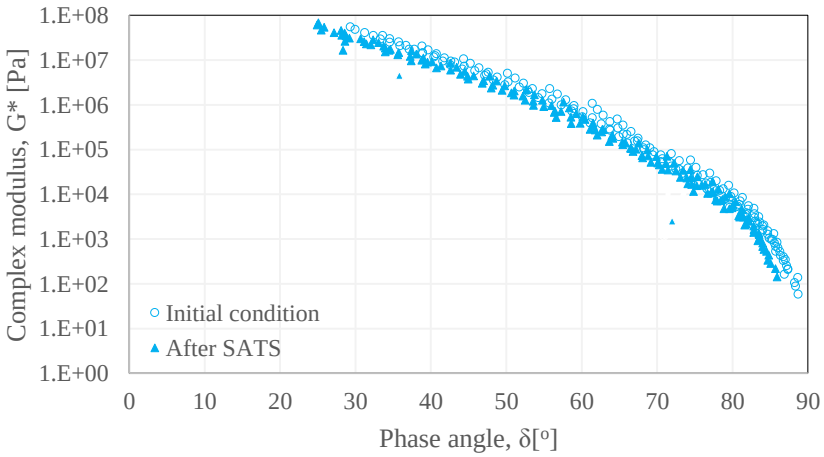


Figure 7.13 Black diagram for Mechanical foamed-RAP 120ºC recovered bitumen

To further evaluate the effects of SATS conditioning on the rheology of all binders, three different rheological indexes were calculated from the data provided by the frequency and temperature sweep tests according to the methodology described in the literature review in Chapter 2: *R*-value, crossover frequency and Glover-Rowe parameter.

Figure 7.15 displays the *R*-value plotted against the crossover frequency for the recovered binders from all the mixtures, before and after SATS conditioning. On one side, the crossover frequency corresponds to a δ of 45º and can be used to measure the hardness of the binders (Booshehrian et al. 2013; Jacques 2015). Aged binders have a lower crossover frequency than virgin binders due to their increased elastic behaviour. The δ master curve of the more aged binders shifts downward, and consequently the δ of 45º is obtained for lower values of crossover frequency. In this sense, all the binders exhibit lower crossover frequency values after SATS conditioning, thus indicating hardening of the binders. On the other hand, *R*-value is related to the shape of the master curve and the oxidation of the bitumen (Booshehrian et al. 2013; Jacques 2015). Its values are commonly high for oxidised binders due to their low crossover frequency values; as the crossover frequency decreases, the difference between the glassy modulus and log $|G^*|$ at the crossover frequency (*R*-value) increases. Therefore, all the binders exhibit high *R*-values after SATS conditioning, thus indicating an increase in the oxidation of the binders. These results support the rheological analysis obtained from the master curves of an overall increase in the stiffness and elastic response of the recovered binders after SATS conditioning.

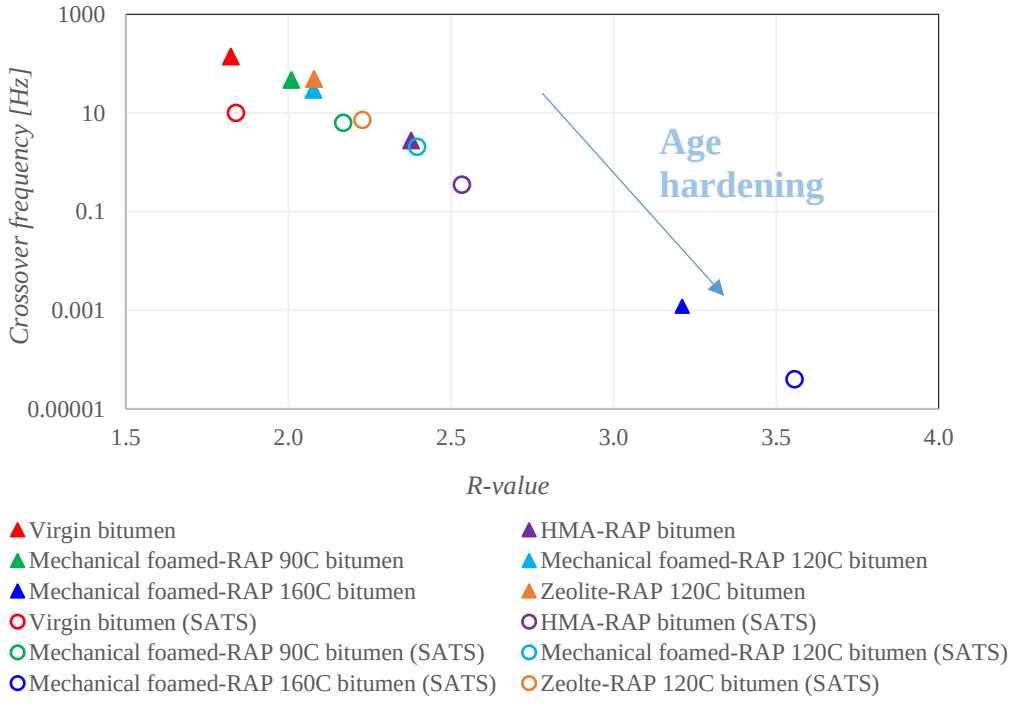


Figure 7.14 Crossover frequency and *R*-value for recovered binders before and after SATS test

Furthermore, the Glover-Rowe (G-R) analysis in Figure 7.16, allows evaluation of the overall rheological changes experienced in the binders after SATS to be evaluated in the black space. In this figure, the virgin binder at the initial condition (before SATS test) is at the bottom right of the plot, as expected, with high δ and low *G-R* values. When the 50% RAP material is incorporated, all binders move towards the upper left of the plot, towards areas where age hardening is potentially occurring, and at higher production temperature of the mixtures the binders keep moving towards the upper left of the plot. These differences in the rheological properties of the RAP binders imply that short term ageing might be occurring during production of the mixtures, but also that the interaction between the aged bitumen from RAP and the virgin bitumen, is different with production temperature of the mixtures, as was observed in the $|G^*|$ master curves in Figure 7.3. It is interesting to see that the binders recovered from the Mechanical foamed mixtures manufactured at 90 and 120ºC, and the Zeolite-RAP mixture manufactured at 120ºC are within the same area of the plot, indicating that the rheological changes occurring in these binders are similar at production temperatures of 90 and 120ºC, regardless of the production technology.

At the frequency value of 0.005 rad/s, from which the G-R parameter is determined, the G-R gives a boundary value of 0.18MPa, which is considered the onset of cracking while a boundary value of 0.45MPa or higher is suggested as an indicator of significance cracking (Jacques et al. 2015). Therefore, this representation indicates that at the initial condition all the recovered binders, except those of the RAP mixtures that were manufactured at 160ºC (i.e. HMA-RAP bitumen and Mechanical foamed-RAP 160C bitumen) pass the onset cracking criteria. These results indicate that the addition of 50% RAP content at 160ºC, accelerates the ageing susceptibility of the bitumen, which could lead to cracking problems. For the same RAP content, and lower production temperatures of 90 and 120ºC instead, the increase in stiffness could be considered positive for rutting. After SATS conditioning, the recovered binders move closer to the onset of cracking. Furthermore, the recovered binder from the mechanical foamed-RAP mixture manufactured at 160ºC, exhibits the worst performance as shown by the highest R-value and the lowest cross-over frequency, compared to the rest of the recovered binders at the initial condition (before SATS test).

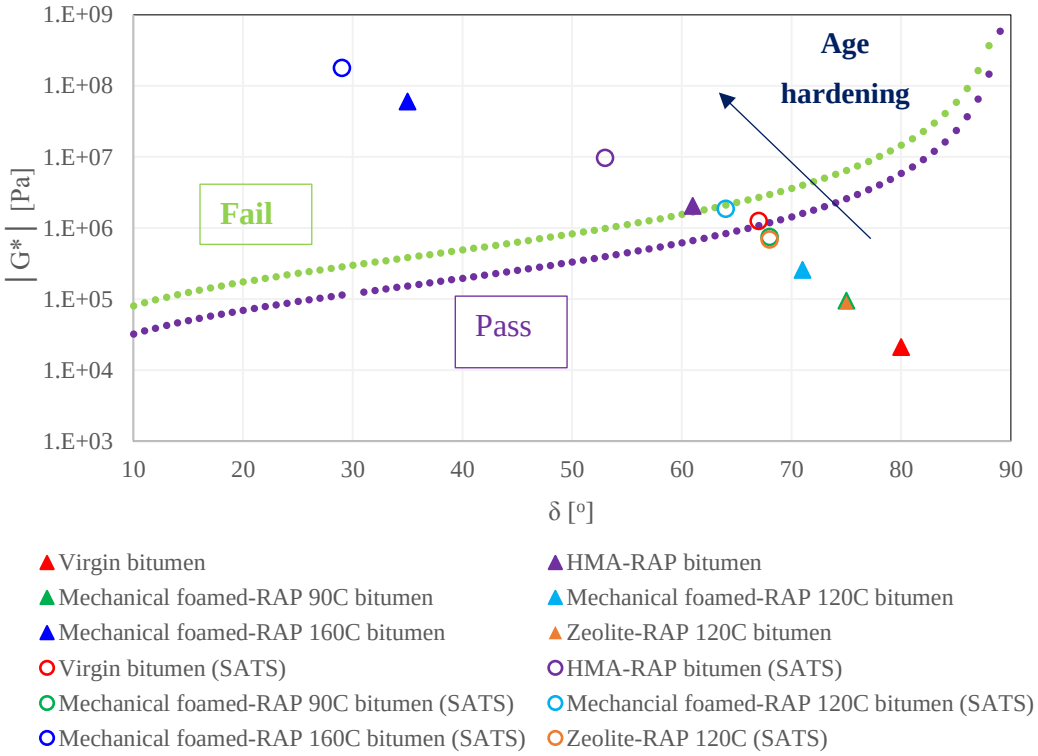


Figure 7.15 DSR-Glover-Rowe parameter for recovered binders before and after SATS test at 15ºC and 0.005 rad/sec

In order to quantify the differences in the rheological properties of the binders recovered from the FBM-RAP mixtures, the differences in the G-R parameter before and after SATS conditioning were calculated and displayed in Table 7-6.

Table 7-6 G-R parameter values for all recovered binders

Bitumen	G-R parameter		Difference	Ratio
	Initial condition	After SATS	[%]	
Virgin bitumen	2.11E+04	1.26E+06	5864	59.6
Mechanical foamed-RAP 90C bitumen	9.45E+04	7.51E+05	694	7.9
Zeolite-RAP bitumen	9.04E+04	6.86E+05	659	7.6
Mechanical foamed-RAP 120C bitumen	2.58E+05	1.86E+06	621	7.2
HMA-RAP bitumen	2.06E+06	9.74E+06	374	4.7
Mechanical foamed-RAP 160C bitumen	6.02E+07	1.80E+08	199	3.0

Data in Table 7-6 provides the same findings obtained from the rheological analysis of the binders (e.g. ageing indexes) of lower differences in the rheological properties before and after SATS conditioning for the stiffer binders. For instance, the virgin bitumen exhibited the highest differences in G-R parameter due to the soft consistency of this bitumen compared to the binders recovered from the RAP containing mixtures where an already aged binder is incorporated, thus implying that the latter binders are less susceptible to age hardening. Indeed the differences in the rheological properties for both conditions (before and after SATS conditioning) are lower for binders recovered from mixtures that were manufactured at the highest temperatures of 160^{o}C , than those recovered from mixtures that were manufactured at the lower production temperatures of 90 and 120^{o}C, thus corroborating the fact that the stiffer binders experienced less hardening effects.

7.5 Conclusions

- In terms of the SATS testing protocol for FAM materials, employing the initial standard test which includes moisture conditioning process at high temperature (i.e. 85^{o}C) and high pressure (i.e. 2.1 MPa) for 65h, is still not enough to achieve high level of saturation in the FAM materials, and evidence damage occurring in these materials by the presence of moisture.

- In terms of the production temperature and at the initial condition of the mixtures (i.e. before SATS conditioning), increasing the production temperature from 90 to 120^{o}C was found to have comparable properties to those of the Virgin HMA. However, manufacturing a mixture with high-RAP content at 160^{o}C increases the overall stiffness and elastic response of the mixtures.

- In general, the RAP-FBMs experienced age-hardening after SATS conditioning as seen by an overall increase in their stiffness and elastic response after the conditioning process. The composition of the FAM materials (i.e. rich bitumen phase and small size air voids) are believed to restrain the intrusion of water, leading to major effects generated by the presence of high temperatures (i.e. ageing) in an environment where both high moisture and temperature are present. Furthermore, in terms of the degradation effects of water, these materials did not show a decrease in their mechanical capacity (i.e. $|G^*|$) under the presence of this agent, indicating a low susceptibility to moisture damage. These results are considered positive, highlighting the potential of these materials to restrain water infiltration.

- In terms of the production temperature in combination with 50%RAP material, a mixing temperature of 160ºC significantly increased the initial stiffness of the FAM mixtures, however after SATS conditioning the resultant mixtures exhibited less age-hardening effects. Lower production temperatures of 90 and 120ºC, resulted in mixtures with comparable properties to that of the Virgin HMA, however the ageing effects in these mixtures are more significant. The initial increase in G* at high production temperatures of 160ºC could be considered positive in areas where rutting in asphalt mixtures is the main concern in terms of performance. However, in areas where fatigue and low temperature cracking are the main phenomena of interest, additional increase in stiffness of the mixtures could be a major concern.

- The rheological properties of the recovered binders at the initial condition corroborated various behaviours observed in the corresponding FAM mixtures. For instance, results indicated that the blend between the hard (RAP) binder and the soft virgin binder in the HMA-RAP mixture did not result in a blend with similar penetration grade to that of the bitumen present in the Virgin HMA, as was theoretically assumed. In addition, the increased hardness and elastic behaviour of the bitumen recovered from the Mechanical foamed-RAP mixture explained the increased stiffness observed in the corresponding FAM mixture.

- The different stiffness of the recovered binders validated that the degree of blending in the RAP mixtures was a function of the production temperature and production process used to manufacture the FAM mixtures.

- Dynamic shear rheometry on binders recovered from cores after they were subjected to the SATS protocol showed that the bitumen had been significantly aged by the conditioning process compared to the initial condition, and as seen in their corresponding FAM mixtures the age-hardening effects were higher in the binders that presented lower initial stiffness.

- Overall as seen in the analysis of the master curves, after SATS conditioning the binders' stiffness and elastic response increased. In this regard, crossover frequency decreases and R-value increases with SATS conditioning, thus indicating an increase in the overall hardness and oxidation of the binder.

Chapter 8 : Summary, Conclusions and Recommendations for Future Research

8.1 Summary

The research in this thesis looked at three main areas:

- The effects of foaming technology and production temperatures (i.e. 90, 120 and 160ºC) on the rheological characteristics of foamed bitumen mixtures manufactured by the traditional method of cold water injection into hot bitumen, known as mechanical foaming, and by a regular WMA technology which includes the incorporation of zeolites.
- The effects of RAP material and production temperature (i.e. 90, 120 and 160ºC) on the rheological characteristics of the produced asphalt mixtures by means of mechanical foaming, and by the incorporation of zeolites.
- The combined effects of moisture and oxidative ageing on the rheological response of the RAP-FBMs.

The two foaming technologies evaluated have been designed for different applications. For instance, the mechanical foaming technology has been intended to be used mainly for cold processes, or cold recycling applications, whereas the zeolite foaming technique, has been intended to be used mainly for warm mix asphalt production. This research evaluates a slightly different way of using the foamed bitumen produced with both technologies, which includes the extended applications of both technologies for half-warm (mixing temperatures of 90ºC), warm (mixing temperatures of 120ºC), and hot applications (mixing temperatures of 160ºC), and the incorporation of RAP material in these mixtures, which constitutes one of the novelties of this research.

The advanced material characterisation was conducted at the meso-scale (i.e. not using full composition asphalt mixture specimens but specimens representing the fine aggregate matrix (FAM) or asphalt mortar present in the full mixtures). Specifically, FAM is herein defined as a combination of bitumen, fine aggregates (below 1mm) and air voids. The use of this type of mixture provides an insight of the behaviour of full-scale asphalt mixtures (Chapter 2), in addition it has been recognised that most damage processes, specially fatigue cracking and moisture damage, initiate or propagate through this matrix (Chapter 2). The research was therefore built up in five stages, starting with the

design of the FAM mixtures in Chapter 3, based on the design of a full scale surface course asphalt mixture, followed by the selection of the appropriate testing methodology for FAM materials to be used with the Kinexus Dynamic Shear Rheometer (DSR) (Chapter 3). Then, the rheological testing of the produced FAM mixtures with two foaming technologies was undertaken in Chapter 4, and of the produced FAM mixtures after replacing 50% of the components with RAP material, in Chapter 5. Results from the mixtures produced by means of the mechanical process in Chapter 4, showed that mixing process and production temperatures are critical to the rheological characteristics of the mixtures. For the zeolite-containing mixtures, the incorporation of the foaming additives did not generate significant changes in the rheological properties of the final mixtures over the different production temperatures evaluated. When RAP material was incorporated into these mixtures in Chapter 5, the rheological response of the mixtures were found to be a function of the foaming technology (i.e. particularly mixing) and production temperature of the mixtures. At last, durability of the RAP-FBMs to resist moisture and oxidative ageing was assessed through rheological testing in Chapters 6 and 7. The laboratory conditioning protocols used in the thesis, namely BS EN 12697-12:2008, including further conditioning in a water bath at 60ºC for 6, 15 and 30 days (Chapter 6), and the Saturating Ageing Tensile Stiffness (SATS) adapted for the fine-RAP-FBMs (Chapter 7), which constitutes another novel aspect of this research, demonstrated that in the presence of moisture and high temperatures the age-hardening related effects dominate the response of these materials.

The Dynamic Mechanical Analysis (DMA) testing procedure was identified as a suitable technique to conduct the advanced rheological characterisation of the produced FAM mixtures. DMA is a non-standardised method that has been successfully used to characterise linear and non-linear viscoelastic properties, and deterioration processes of FAM materials that are present in regular HMA mixtures (Chapter 2). Although this method has become popular to characterise the fine matrix of HMA or certain WMA mixtures, no work has been reported to date on their use to evaluate different FBM materials and, even less, on mixtures combining FBM and RAP materials, which constitutes another novel aspect of this study. Besides, the application of this technique has enabled the evaluation of different variables, which are related with construction processes that can influence the performance of the produced mixtures. These variables include the production methods (i.e. foaming technologies), production temperatures, and environmental effects such as the presence of moisture and elevated temperatures.

The FAM materials evaluated in this study, did not exhibit a rheologically simple behaviour, increasing the complexity of the rheological analysis. Constructing rheological master curves of the viscoelastic properties using the same shifting factors used to construct the shear complex modulus

master curve (for example) requires special attention when interpreting the time and temperature dependency of the materials. For this reason, the effect of data manipulation (i.e. shifting procedure) must be considered. When evaluating the rheological characteristics of these materials, it is important to consider various forms of rheological data representation, such as isochronal plots at different frequencies, and/or the black diagram curves. The latter, which provides the complete rheological behaviour of the material ($|G^*|$ and δ), eliminating the frequency and temperature from the plot with no need to perform any shifting of the measured data or relying on the FAM materials fulfilling the TTSP.

Furthermore, with the sensitivity of the foamed bitumen mixtures with production process, undertaking rheological testing over wide temperature and frequency ranges by means of the DMA testing procedure, allows any alterations in the mixture design to be determined. The DMA testing procedure is also sensitive to the material properties making it a useful tool for this purpose.

8.2 Conclusions

The main conclusions that can be drawn from the rheological research undertaken in this thesis are:

- The rheological characteristics of the Mechanical foamed mixtures strongly depend on the production process and the mixing temperature of the materials. Conversely, the rheological characteristics of the zeolite containing mixtures are not significantly affected by changes in the production temperature. Thus, the foaming technology and production temperature, have different impacts on the produced mixtures and should be considered when manufacturing these type of mixtures.

- The incorporation of 50% RAP material into zeolite containing FBMs generated mixtures with comparable properties to those of a reference Virgin HMA (no RAP material), at various production temperatures. The incorporation of 50% RAP material into FBMs produced by means of the Mechanical foaming process, generated a different rheological response with respect to the reference Virgin HMA, with production temperature. Thus the incorporation of 50%RAP material has different effects on the produced mixtures, which can be partially due to different degrees of blending between old-new materials with different final temperatures of the mixtures, and the production processes (particularly mixing) or foaming technology employed.

- In the presence of moisture and high temperature environment, the age-hardening related effects dominate the response of the FAM materials. The rich bitumen phase, the small-sized voids along with the high quality of the aggregates (i.e. limestone) used for the preparation of these materials, make it difficult for the water to reduce their mechanical capacity, whereas high temperatures certainly generate some effect on the rheological properties of the materials, which consist of a shifting of the rheological characteristics to a more elastic behaviour with respect to their initial (dry) condition.

More specifically, with respect to the influence of the foaming technique and production temperature the following were the main findings:

- Low production temperatures of 90 and 120ºC by means of the mechanical foamed process generated mixtures with unexpected increased stiffness. Poor bitumen dispersion at these reduced mixing temperatures and lower effective binder content were the main reasons associated with this response.

- Producing FBMs by means of the mechanical foaming process for hot applications (i.e. aggregates heated at 160ºC) generates a mixture with similar rheological properties of a conventional HMA mixture. This means that the application of this foaming technology for hot applications does not offer any advantage compared to the traditional HMA.

- The effect of incorporating a certain amount of heat into the aggregates for further combination with the hot foamed bitumen (laboratory scale) was found to aid mainly in the bitumen dispersion within the mixture. This indicates that the general dependency of the mechanical foamed mixtures characteristics to mixing process should be considered before its application to ensure that a stable asphalt mixture is produced meeting the design requirements.

- FBMs produced by means of zeolite foaming technologies were not sensitive to the production temperature of the mixtures.

- Increasing the mixing temperature of the mixtures, does not generate an increase in the dynamic shear modulus of the bitumen present in the Mechanical foamed mixtures, while a slight increase in the dynamic shear modulus of the bitumen present in the zeolite containing mixtures is

generated. These effects are linked to the material preparation requirements for each foaming technology.

Further conclusions obtained from the characterisation of the rheological properties of the foamed mixtures produced by means of both foaming techniques containing 50% of RAP were:

- The incorporation of 50% RAP material into the Zeolite-FAM mixtures, were not significantly sensitive to the production temperature. Mixtures with comparable properties to that of a reference Virgin HMA, at mixing temperatures as low as 90ºC or as high as 160ºC, in the laboratory scale, can be produced. Thus replacing 50% of the virgin components of the foamed bitumen mixtures manufactured with this foaming technology by RAP material and employing mixing temperatures as low as 90ºC constitute a promising alternative to generate a mixture with comparable properties to the traditional HMA, obtaining energy savings and contributing to the conservation of natural resources (as old materials can be reused).

- The incorporation of 50% RAP material into the mechanical foamed mixtures using low mixing temperatures of 90 and 120ºC, generated mixtures with comparable properties to that of the reference Virgin HMA, at a laboratory scale. This response is contrasted by the incorporation of 50%RAP into the mechanical foamed mixtures when the mixing temperature is increased to 160ºC. This indicates that the final properties of these mixtures are, to some extent, sensitive to the production process and mixing temperatures employed.

Further conclusions from the environmental conditioning protocol by means of BS EN 12697-12:2008, including further conditioning in a water bath at 60ºC at different conditioning times on the rheological response of the RAP-FBMs were:

- Submerging the RAP-FBMs in a water-soaking regime at 60ºC for up to 30 days conditioning time generated a modification in the $|G^*|$ values of the mixtures, with a general increase in $|G^*|$ with conditioning time being the most dominant response. These changes are associated with oxidative ageing and hardening of the bitumen while present in the water bath at 60ºC. The Mechanical foamed-RAP mixtures presented higher increase in $\left|G^*\right|$ than the Zeolite-RAP technology mixtures.

- The changes in the dynamic shear modulus of the RAP-FBMs after the environmental conditioning process, and after pure ageing conditioning (i.e. no presence of moisture) were to generate a general increase in the dynamic shear modulus of the mixtures with the age-hardening effects after the pure-ageing being more significant, as can be expected. The magnitude of increase in the dynamic shear modulus in both conditioning processes, for the various RAP-FBMs mixtures evaluated, was sensitive to the type of foaming technology used and the production temperature of the mixtures.

- The mixtures with higher initial stiffness exhibited the lowest ageing ratios, meaning that the ageing susceptibility is sensitive to the initial properties of the materials.

Further conclusions from the combined effects of ageing and moisture on the rheological response of the RAP-FBMs by means of SATS conditioning protocol on FAM materials include:

- Under dry conditions, the production of a mechanical foamed-RAP mixture at 160ºC exhibited consistently a significant increase in the dynamic shear complex values with respect to the reference Virgin HMA. Similar behaviour was obtained for the reference HMA-RAP mixture manufactured at 160ºC (with no foaming technology). Binders recovered from these mixtures indicated not only that the blend between the hard (RAP) binder and the soft virgin binder did not result in a similar penetration as the bitumen present in the Virgin HMA as was theoretically assumed, but reflecting the influence of other factors during the production process at this elevated temperature of 160ºC that generate a significant increase in the stiffness of these mixtures.

- The combined effects of moisture and high temperature during SATS conditioning generated a stiffening effect and an increased elastic response on the evaluated RAP-FBMs, as indicated by an increase in their dynamic shear complex modulus values and a decrease in their phase angle values respectively, with respect to their initial condition. The high quality of the constitutive phases of the mixtures (i.e. limestone aggregates) along with the composition of these materials (i.e. rich bitumen phase) and small air voids are the main reasons of these behaviour.

- Binders recovered from the RAP-FBM fine mixtures after SATS conditioning, showed an overall hardening effect and an increased elastic response. These effects of oxidative ageing after SATS conditioning were less severe for the binders having higher initial stiffness.

8.3 Recommendations for future work

The research conducted in this work and its findings suggest the following avenues for the study:

- This study was performed using a specific procedure to manufacture the mixtures in the laboratory. In the case of the mechanical foamed mixtures, a Hobart type mixture with a flat type agitator was adapted to the laboratory foamed machine in order to maintain the temperature of the aggregates. Acknowledging past literature that is available on the production of these mixtures particularly for cold applications and half-warm processes, and also as identified in this work, the challenge to produce foamed bitumen mixtures by means of the mechanical foaming process for half-warm, warm and hot applications in the laboratory means that studying different mixing processes to produce these type of mixtures for these extended applications is recommended. A further approach may include the use of a twin shaft heated mixer. In the case of the zeolite containing mixtures, the rheological properties were not sensitive to the production temperature. As was already described, it is hypothesised that in the zeolite-bitumen pre-blending process, the foaming action is lost before this blend is incorporated into the aggregates making this procedure similar to just mixing asphalt mixtures at different temperatures. Therefore, it is worth studying alternative forms of incorporating the zeolites into the asphalt mixtures (e.g. adding the zeolites to the asphalt mixture at the same time as the binder, as seen in the literature review in Chapter 2) or increasing the amount of zeolite to be incorporated.

- In this research, the level or degree of blending between the old bitumen from RAP and virgin bitumen was found to be mainly a function of the foaming technology (particularly mixing), and the mixing temperature of the mixtures. At a mixing temperature of 160^{o}C for the Mechanical foamed-RAP an unexpected significant increase in the dynamic shear modulus was obtained. This breaks the concept of complete blending, reflecting that other factors are contributing in the final properties of this mixture, which are not well understood. Therefore, further research to better understand this behaviour is needed.

- In this study the principal objectives of the environmental conditioning and the SATS conditioning protocol employed, were to evaluate the combined effects of ageing and moisture sensitivity on the rheological response of the RAP-FBMs. A general increase in the dynamic shear modulus of the mixtures in both conditioning protocols was observed, suggesting that the age-hardening effects due to oxidative ageing dominate the response of these materials over the

degradation effects that can be generated by the presence of moisture. Additional research is needed to understand the degradation effects due to moisture within these materials. It is therefore recommended to supplement the rheological response measured in this research with fundamental properties that affect physical adhesion between the bitumen and aggregate and internal mastic cohesion and the propensity to lose these bonds in the presence of water. For instance, the principles of Surface Free Energy (SFE) used in combination with thermodynamic theory would allow the theoretical adhesive bond quality between recovered binders from the RAP-FBM fine mixtures and virgin aggregates to be quantified. This methodology would allow the most favourable combinations of bitumens to produce high adhesion values to be determined but also evaluate if these conditions are easily deteriorated or not by the presence of water. These results can be correlated with the results provided after conditioning the samples with both protocols.

- The moisture effects on the RAP-FBMs were evaluated on mixtures produced with limestone aggregates, which are well known for their resistance to moisture damage. A future approach to moisture effects would be to extend this research including other types of aggregate such as granite, with different physical, chemical and mechanical properties from limestone that can develop a different situation, which is worth studying.

- The shape, size and distribution of the air voids within the asphalt mixtures have an effect on their moisture damage susceptibility, as described in Chapter 2. Therefore studying the distribution and structure of air voids in the produced FAM specimens would provide further information of this phenomenon within these materials. For instance, FAM specimens before and after the environmental and the SATS conditioning protocols can be studied using X-ray computed tomography (CT) technique.

- The current study was based on the linear viscoelastic material properties of the produced mixtures. Further research is needed to evaluate their fracture characteristics and permanent deformation behaviour, which can be performed by means of torsional oscillatory shear tests in the DMA. Specifically for the former, strain-controlled torsional shear time sweep tests can be conducted on the FAM specimens by applying strain levels greater than the satisfactory linear viscoelasticity to generate damage in testing specimens and evaluate their fatigue resistance. For the latter, creep-recovery tests can be conducted to determine the amount of creep strain and

irrecoverable strain of individual FAM mixtures subjected to different strain levels at high temperatures, in order to evaluate their permanent deformation characteristics.

- On the basis of satisfactory performance in the laboratory, practical validations are recommended to verify the behaviour on pavement performance under real service conditions of foamed bitumen mixtures manufactured by means of mechanical foaming and by the incorporation of zeolites with various mixing temperatures, and their behaviour in the production of mixtures containing high RAP content (i.e. 50%). It is recommended to conduct field validations and compare with laboratory prepared specimens.

Based on the observations and conclusions obtained in this study, the following are the main recommendations regarding the use of these materials in paving works:

- In terms of the FBMs, the high dependence of the rheological response of these mixtures on the foaming technology employed (i.e. particularly mixing process), mixing temperature and preparation of the materials, means that these variables need to be optimised to guarantee that the mixtures meet the design requirements. It is important to recognize that the control of these parameters might not be accurately possible in field but it is important to understand the impacts of these parameters in the mix formula and therefore on the properties of the asphalt mixtures. For the mechanical foaming technique this optimisation should be directed on the implementation of a high quality mixer that is able to maintain the temperature of the aggregates during the mixing process. As was already described, a heated twin-shaft pug mill is highly recommended. In field, this system would require the systematic extraction of samples for a binder content check to verify mix design requirements. In the case of implementing these mixtures, incorporating a certain amount of heat into the aggregates prior mixing with the foamed bitumen results in mixtures with comparable properties to traditional HMA. Particularly, in this study it was observed that a mixing temperature of 160ºC generated a mixture with similar properties to those of the reference Virgin HMA, making it possible to be applied as part of surface course mixture applications. However, it is noteworthy to mention that energy savings are no longer applicable. It is also important to mention that in this research, an investigation into some of the parameters related to FBMs has been conducted. However, FBMs are complex materials that involve various parameters, thus there is plenty of scope to fully understand the behaviour of FBMs and to predict the performance as accurately as possible.

- For the zeolite foaming technologies, the rheological properties of these mixtures were not sensitive to the mixing temperatures. It was found that the incorporation of zeolites did not significantly increase or decrease the mechanical capacity of the asphalt mixtures with mixing temperature. However they exhibited a slight decrease in stiffness when compared to the reference Virgin HMA. It is therefore recommended to perform field validations to evaluate the long term performance of these materials, and until what extent they can be applied in high quality paving projects. It is important to emphasize that these mixtures were manufactured using specific set of materials (i.e. aggregates and bitumen) and WMA additives, therefore restraining the possibility of providing generalised conclusions on the overall performance of the evaluated foaming technologies.

- In terms of the FBMs containing RAP material, it was apparent that the production parameters, such as mixing process, and mixing temperature have a significant impact on the mixtures performance. Particularly, these parameters influence the level of blending between RAP and virgin binders and therefore the properties of the resultant mixtures. All Zeolite-RAP mixtures evaluated exhibited comparable properties to that of the reference Virgin HMA, while two out of three Mechanical foamed-RAP mixtures evaluated exhibited comparable properties to those of the reference Virgin HMA. These results suggest that incorporating high amounts of RAP material (e.g. 50%) is a promising alternative to generate mixtures with comparable properties to that of a Virgin HMA. These results are considered positive since they suggest that it is possible to produce high quality mixtures for paving applications incorporating high contents of RAP material and in combination with foaming technologies. Furthermore, the use of a soft virgin bitumen mitigates the overall hardness and elastic properties of the hard RAP bitumen. It is important to mention that before the implementation of these materials in a particular project, a detailed study that involves the design of the mixture and its respective characterisation is required.

- It is worth mentioning that in this study the properties of the RAP material were controlled, which represents the ideal material to be reclaimed in asphalt mixtures. However, in practice the properties of the RAP material might be unknown and the variability of its properties restrains the possibility of providing generalised conclusions on the overall rheological and mechanical performance of mixtures containing RAP material. It is therefore recommended to perform some field validations to confirm the performance of the material on site.

References

BS 2000-49:2007. Bitumen and bituminous binders – determination of needle penetration.

BS 2000-58:2007. Bitumen and bituminous binders – determination of the softening point – ring and Ball method.

BS 2000-80:2007. Bitumen and bituminous binders – determination of the Fraass breaking point.

BS 2000-505:2010. Bitumen and bituminous binders – determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus.

BS EN 12697-4:2005. Bituminous mixtures-Test methods for hot mix asphalt-Part 4: Bitumen recovery: Fractioning column.

BS EN 12697-35:2016. Bituminous mixtures — Test methods Part 35: Laboratory mixing.

EN 13108-8:2005. Bituminous mixtures – material specifications – Part 8: Reclaimed asphalt.

ABEL, F. & HINES, C. R. 1979. Base stabilization with foamed asphalt. . *Report No. CDOH-SMB-R-79 (FHWA-CO-RD-79-5).* Denver, Colorado: Colorado Division of Highways.

ACADEMY, A. 2009. Bitumen Stabilised Materials: A Guideline for the Design and Construction of Bitumen Emulsion and Foamed Bitumen Stabilised Materials in Technical Guidelines.

ACOTT, S. M. 1979. Sand stabilization using foamed bitumen. . *Third Conf. Asph. Pavements South. Africa, Durban.*

AIREY, G. D. 1997. Rheological characteristics of polymer modified and aged bitumens. The University of Nottingham.

AIREY, G. D. 2010. Bituminous Materials. *In:* DOMONE, P. & ILLSTON, J. (eds.) *Construction Materials their nature and behaviour* 4th edition Chapter 27 - 32 ed. Great Britain.

AIREY, G. D., CHOI, Y. K., COLLOP, A. C., MOORE, A. J. V. & ELLIOTT, R. C. 2005. Combined laboratory ageing / moisture sensitivity assessment of high modulus base asphalt mixtures. *Journal of the Association of Asphalt Paving Technologists,* 74**,** 307-346.

AIREY, G. D. & HUNTER, A. E. 2003. Dynamic mechanical testing of bitumen: sample preparation methods. *Proceedings of the Institution of Civil Engineers***,** 85-92.

AL-QADI, I., ELSEIFI, M. & CARPENTER, S. H. 2007. Reclaimed Asphalt Pavement - A literature review. Urbana, IL.

ALJUBORYL, A., AIREY, G. D. & GRENFELL, J. 2017. Laboratory evaluation of Stiffness and Fatigue Susceptibility of Asphalt Paving Materials *Bearing Capacity of Roads Railways and Airfields*.

ALOSSTA, A. A., ZEIADA, W. A. & KALOUSH, K. E. 2011. Evaluation of warm mix asphalt versus conventional hot mix asphalt for field and laboratory compacted specimens *Journal of the Transportation Research Board*.

ANDERSON, D. A., CHRISTENSEN, D. W., BAHIA, H. U., DONGRE, R., SHARMA, M. G., ANTLE, C. E. & BUTTON, J. 1994. Binder characterization and evaluation. Volume 3: Physical Characterization. *SHRP-A-369.* Washington, DC: Strategic Highway REsearch Program, National Research Council.

AUSTROADS 2015. Maximising the Re-Use of Reclaimed Asphalt Pavement: Outcomes of Year Two: RAP Mix Design. AP-T286-15. . Sydney, NSW.: Austroads.

BARNES H A, H. J. F. A. W. K. 1989. An introduction to rheology. Netherland: Elsevier Science Publishers.

BELL, C. A. 1989. Summary report on aging of asphalt-aggregate systems. Oregon State University.

BONAQUIST, R. 2005. Laboratory evaluation of hot mix asphalt (HMA) mixtures containing recycled or waste product materials using performance testing.

BONAQUIST, R. 2011. Mix design practices for warm mix asphalt. Washington, D.C.: Transportation Research Board.

BONAQUIST, R. & CHRISTENSEN, D. W. 2005. Practical Procedure for Developing Dynamic Modulus Master Curves for Pavement Structural Design. . *Transportation Research Record No. 1929***,** pp. 208–217.

BOOSHEHRIAN, A., MOGAWER, W. S. & BONAQUIST, R. 2013. How to Construct an Asphalt Binder Master Curve and Assess the Degree of Blending between RAP and Virgin Binders. *Journal of Materials in Civil Engineering* 25**,** 1813-1821.

BOWERING, R. H. & MARTIN, C. L. 1976. Foamed bitumen production & application of mixes evaluation & performance of pavements. *Proceedings of the Association of Asphalt Paving Technologists (AAPT),* 45**,** 453–477.

BUTTON, J., ESTAKHRI, C. & WIMSATT, A. 2007. A Synthesis of Warm Mix Asphalt. College Station, Texas: Texas Transportation Institute.

CARO, S., BELTRÁN, D. P., ALVAREZ, A. E. & ESTAKHRI, C. 2012. Analysis of moisture damage susceptibility of warm mix asphalt (WMA) mixtures based on Dynamic Mechanical Analyzer (DMA) testing and a fracture mechanics model. *Construction and Building Materials,* 35**,** 460-467.

CARO, S., MASAD, E., AIREY, G., BHASIN, A. & LITTLE, D. 2008a. Probabilistic Analysis of Fracture in Asphalt Mixtures Caused by Moisture Damage. *Transportation Research Record: Journal of the Transportation Research Board,* 2057**,** 28-36.

CARO, S., MASAD, E., BHASIN, A. & LITTLE, D. N. 2008b. Moisture susceptibility of asphalt mixtures, Part 1: mechanisms. *International Journal of Pavement Engineering,* 9**,** 81-98.

CASTELO BRANCO, V. T. F. 2008. *A Unified Method For The Analysis Of Nonlinear Viscoelasticity And Fatigue Cracking Of Asphalt Mixtures Using The Dynamic Mechanical Analyzer.* Texas A&M University.

CHRISTENSEN, D. & ANDERSON, D. 1992. Interpretation of Dynamic Mechanical Test Data for Paving Grade Asphalt. *Journal of the Association of Asphalt Paving Technologists,* Vol. 61**,** pp. 67–116.

CHRISTOPHER JACQUES, JO SIAS DANIEL, THOMAS BENNERT, GERALD REINKE, AMIRHOSSEIN NOROUZI, CHRISTOPHER ERICSON, WALAA MOGAWER & KIM, Y. R. 2015. Effect of Silo Storage Time on the Characteristics of Virgin and Reclaimed Asphalt Pavement Mixtures. *TRB 95th Annual Meeting Compendium of Papers. Transportation Research Board***,** 16-1474.

COLLOP, A. C., CHOI, Y., AIREY, G. D. & ELLIOT, R. C. 2004. Development of the Saturation Ageing Tensile Stiffness (SATS) Test. *ICE Journal of Transport,* 157**,** 163-171.

COPELAND, A. 2011. Reclaimed Asphalt Pavement in Asphalt Mixtures: State of the Practice. McLean, VA: Federal Highway Administration.

CORRIGAN, M. 2008. Warm mix asphalt technologies and research. *Federal Highway Administration-FHWA.*

CSANYI, L. H. 1957. Foamed asphalt in bituminous paving mixtures. *Highway Research Board Bulletin***,** 108-122.

D'ANGELO, J., HARM, E., BARTOSZEK, J., BAUMGARDNER, G., CORRIGAN, M., COWSERT, J., HARMAN, T., JAMSHIDI, M., JONES, W., NEWCOMB, D., PROWELL, B., SINES, R. & YEATON, B. 2008. Warm Mix Aspahlt European Practice.

DANIEL, J., POCHILY, J. & BOISVERT, D. 2010. Can more reclaimed asphalt pavements be added? Study of extracted binder properties from plant produced mixtures with up to 25% reclaimed asphalt pavement. *Transportation Research Record: Journal of the Transportation Research Board*
2180**,** 19–29.

DE SOUSA, P. C. 2010. *Automated Protocol For Analysis of Dynamic Mechanical Analyzer Data From Fine Aggregate Asphalt Mixes.* Texas A&M University.

DICKINSON E. J. , A. W. H. P. 1974. The Dynamic Shear Modulus of Paving Asphalts as a Function of Frequency. *Transactions of the Society of Rheology,* 18 (4)**,** 591-606.

EAPA 2008. Arguments to stimulate the government to promote asphalt reuse and recycling. *In:* ASSOCIATION, E. A. P. (ed.). Brussels, Belgium.

ENGELBRECHT, D. 1999. Manufacturing foam bitumen in a standard drum mixing asphalt plant. *7th Conf. Asph. Pavements South. Africa***,** 1–8.

EPPS, A., ARAMBULA, E., YIN, F., CUCALON, L. G., CHOWDHURY, A., LYTTON, R., EPPS, J., ESTAKHRI, C. & PARK, E. S. 2014. Evaluation of the moisture susceptibility of WMA technologies. *In:* PROGRAM, N. C. H. R. (ed.). Washington, D.C.

EPPS, J., BERGER, E. & ANAGNOS, J. N. 2003. "Treatments" Moisture Sensitivity of Asphalt Pavements: A National Seminar. *Transportation Research Board.* San Diego, California.

EUROBITUME 1995. First European workshop on the rheology of bituminous binders Brussel: European Bitumen Association.

EUROBITUME 1996. Rheology of bituminous binders Glossary of Rheological Terms: A practical summary of the most common concepts. European Bitumen Association.

FIGUEROA INFANTE, A. S. 2015. *Investigacion sobre el efecto del agua en el asfalto y su impacto en la mezcla asfaltica.* PhD Thesis, Pontificia Universidad Javeriana.

FU, P. 2009. *Micromechanics for Foamed Asphalt Stabilized Materials.* . University of California Davis.

GLOVER, C. J., DAVISON, R. R., DOMKE, C. H., RUAN, Y., JURISTYARINI, P., KNORR, D. B. & JUNG, S. H. 2005. Development of a new method for assessing binder durability with field validation (Report FHWA/TX-05/1872-2). . Austin, TX.: Texas Department of Transportation.

GOODRICH, J. L. 1988. Asphalt and polymer modified asphalt properties related to the performance of asphalt concrete mixes. *Proceedings of the Association of Asphalt Paving Technologists,* 57**,** 116-175.
GRENFELL, J., AHMAD, N., AIREY G. D. & ELLIOTT, R. 2012. Optimising the moisture durability SATS conditioning parameters for universal asphalt mixture application. . *International Journal of Pavement Engineering,* 13**,** 433-450.
GRENFELL, J., APEAGYEI, A. K. & AIREY, G. 2015. Moisture damage assessment using surface energy, bitumen stripping and the SATS moisture conditioning procedure. *International Journal of Pavement Engineering,* 16**,** 411-431.
GUDIPUDI, P. & UNDERWOOD, B. S. 2015. Testing and modeling of fine aggregate matrix and its relationship to asphalt concrete mix. *Transportation Research Record: Journal of the Transportation Research Board,* 2507**,** 120-127.
HAJJ, E. Y., SEBAALY, P. E. & SHRESTHA, R. 2007. A laboratory evaluation on the use of recycled asphalt pavements in HMA mixtures. . Reno, NV: Washoe Regional Transportation Commission, University of Nevada.
HANSEN, K. R. & COPELAND, A. 2013. 2nd Annual Asphalt Pavement Industry Survey on Reclaimed Asphalt Pavement, Reclaimed Asphalt Shingles, and Warm-Mix Asphalt Usage: 2009–2011 (IS 138). . Lanham, Maryland.: National Asphalt Pavement Association.
HARVEY, J. T. & TSAI, B.-W. 1994. Effects of Asphalt Content and Air Void Content on Mix Fatigue and Stiffness. *Transportation Research Record*.
HE, Y., ALAVI, M. Z., JONES, D. & JOHNHARVEY 2016. Proposing a solvent-free approach to evaluate the properties of blended binders in asphalt mixes containing high quantities of reclaimed asphalt pavement and recycled asphalt shingles. *Construction and Building Materials,* 114**,** 172-180.
HERNÁNDEZ, S. 2013. *Influencia de la Humedad Relativa en el Comportamiento y el Deterioro de Matrices Asfálticas Finas.* Universidad de Los Andes.
HICKS, G., SANTUCCI, L. & ASCHENBRENER, T. 2003. Introduction and seminar objectives. Moisture sensitivity of asphalt pavements: a national seminar, San Diego, California. . Washington DCTransportation Research Board.
HIGHWAYS-AGENCY. 2007. *Manual of contract documents for Highway works. Vol 1. Specification for Highway works.* Stationery Office, London patent application.
HUNTER, R. N., SELF, A. & READ, J. 2015. *The Shell Bitumen Handbook,* Westminster, London ICE Publishing.
HURLEY, G. C. & PROWELL, B. D. 2005. Evaluation of Aspha-Min® zeolite for use in warm mix asphalt. National Center for Asphalt Technology (NCAT) Auburn University.
IM, S., YOU, T., BAN, H. & KIM, Y.-R. 2015. Multiscale testing-analysis of asphaltic materials considering viscoelastic and viscoplastic deformation. *International Journal of Pavement Engineering*
18**,** 783-797.
JENKINS, K. J. 2000. *Mix Design Considerations for Cold and Half-Warm Bituminous Mixes with Emphasis on Foamed Bitumen.* University of Stellenbosch.
JENKINS, K. J., MOLENAAR, A. A. A., DE GROOT, J. L. A. & VAN DE VEN, M. E. C. 2000. Developments in the uses of foamed bitumen in road pavements. *HERON,* 45.
JONGEPIER, R. & KUILMAN, B. 1969. Characterization of the Rheology of bitumens *Proceedings of the Association of Asphalt Paving Technologists,* 38**,** 98-122.
KANDHAL, P. S. & CHAKRABORTY, S. 1996. Effect of asphalt film thickness on short-and long-term aging of asphalt paving mixtures., Transportation Research Record: Journal of the Transportation Research Board. *Transportation Research Record: Journal of the Transportation Research Board,* 1535**,** pp. 83-90.
KIGGUNDU, B. M. & ROBERTS, F. L. 1988. Stripping in HMA mixtures: state-of-the-art and critical review of test methods. Report 88-02.: National Center for Asphalt Technology Alabama: Auburn University.
KIM, Y. R., D.N. LITTLE & LYTTON., R. L. 2003. Fatigue and Healing Characterization of Asphalt Mixtures. Journal of Materials in Civil Engineering. 15**,** pp. 75-83.
KIM, Y. R. & LITTLE, D. N. 2004. Linear viscoelastic analysis of asphalt mastics. *J. Mater. Civil Eng.***,** 122–132.
KOENDERS, B. G., D.A. STOKER, C. ROBERTUS, O. LARSEN & JOHANSEN, J. 2002. WAM-Foam, asphalt production at lower operating temperatures. . *Proceedings of the Ninth International Conference on Asphalt Pavements. Copenhagen.* .
KUENNEN, T. 2004. Warm Mixes are a Hot Topic. Better Roads. 74-6. James Informational Media. Des Plaines, IA.

KUNA, K., AIREY, G. & THOM, N. 2014. A laboratory mix design procedure for Foamed Bitumen Mixtures (FBM). *Transportation Research Record: Journal of the Transportation Research Board*.

LEE, D. 1973. Asphalt durability correlation in Iowa. *Transportation Research Record,* 468**,** pp. 43-60.

LI, X., CLYNE, T. R. & MARASTEANU, M. O. 2004. Recycled Asphalt Pavement (RAP) Effects on Binder and Mixture Quality. . Minneapolis: Minnesota Department of Transportation.: University of Minnesota, Department of Civil Engineering. .

LIAO, M. C. 2007. *Small and Large Strain Rheological and Fatigue Characterisation of Bitumen-Filler Mastics.* . The University of Nottingham.

LONG, F. M., THEYSE, H. L. & VENTURA, D. F. C. 2004. Characterisation of Foamed Bitumen Treated Material from HVS Test Section. . Pretoria, South Africa.: CSIR Transportek.

LORIA, L., HAJJ, E. Y., SEBAALY, P. E., BARTON, M., KASS, S. & LISKE, T. 2011. Performance Evaluation of Asphalt Mixtures with High Recycled Asphalt Pavement Content

Transportation Research Record: Journal of the Transportation Research Board 72-81.

LYTTON, R. 2005. Adhesive Fracture in Asphalt Concrete Mixtures. *In J. Youtcheff (Ed).*

LYTTON, R. L., UZAN, J., FERNANDO, E. G., ROQUE, R., HILTUNEN, D. & STOFFELS, S. M. 1993. Development and validation of performance prediction models and specifications for asphalt binders and paving mixes (SHRP-A-357). . Washington, DC.

MALLICK, R. B. & EL-KORCHI, T. 2013. *Pavement Engineering Principles and Practice*, Boca Raton, FL.

MARASTEANU, O. & ANDERSON, D. A. 1999a. Improved Model for Bitumen Rheological Characterization. Eurobitume Workshop on Performance Related Properties for Bitumens Binder. .

MASAD, E., CASTELO BRANCO, V. T. F., LITTLE, D. N. & LYTTON, R. 2008. A unified method for the analysis of controlled-strain and controlled-stress fatigue testing. *International Journal of Pavement Engineering,* 9**,** 233-246.

MASAD, E., ZOLLINGER, C., BULUT, R., LITTLE, D. & LYTTON, R. 2006a. Characterization of HMA moisture damage using surface energy and fracture properties. *Asphalt Paving Technology: Association of Asphalt Paving Technologists-Proceedings of the Technical Sessions*.

MASAD, E. A., ZOLLINGER, C., BULUT, R., LITTLE, D. N. & LYTTON, R. L. 2006b. Characterization of HMA moisture damage using surface energy and fracture properties

Journal of the Association of Asphalt Paving Technologists, 75**,** 713-754.

MAUPIN, G. W., DIEFENDERFER, S.D., & GILLESPIE, J.S. 2008. Evaluation of using higher percentages of recycled asphalt pavement in asphalt mixes in Virginia (Final Report VTRC 08-R22). . Charlottesville, VA: Virginia Transportation Research Council.

MCDANIEL, R. & ANDERSON, R. M. 2001. Recommended use of reclaimed asphalt pavement in the superpave mix design method: Technician's manual (NCHRP Report 452). . Washington, DC.

MCDANIEL, R. S., SHAH, A., HUBER, G. A. & COPELAND, A. 2012. Effects of reclaimed asphalt pavement content and virgin binder grade on properties of plant produced mixtures. . *Road Materials and Pavement Design,* 13**,** 161-182.

MCGENNIS, R. B., KENNEDY, T. W. & MACHEMEHL, R. B. 1984. Stripping and moisture damage in asphalt mixtures. Research Report 253-1, Project 3-9-79-253. Austin, Tx.: Center for Transportation Research. Austin: The University of Texas at Austin.

MEDANI, T. O., HUURMAN, M. & MOLENAAR, A. A. A. 2004. On the Computation of Master Curves for Bituminous Mixes. Proceedings 3rd Eurobitume Congress, Vienna, Austria. Vol. 2**,** pp. 1909–1917.

MENSCHING, D. J., ROWE, G. M. & DANIEL, J. S. 2017. A mixture-based Black Space parameter for low temperature performance of hot mix asphalt. *Road Materials and Pavement Design*.

MOGAWER, W., BENNERT, T., DANIEL, J. S., BONAQUIST, R., AUSTERMAN, A. & BOOSHEHRIAN, A. 2012. Performance characteristics of plant produced high RAP mixtures. . *Road Materials and Pavement Design* 13**,** 183–208.

MONISMITH, C. L., ALEXANDER, R.L. AND SECOR, K.E. 1966. Rheological behaviour of Asphalt Concrete. *Proceedings of the Association of Asphalt Paving Technologists,* 35**,** 400-450.

MONTEPARA, A., ROMEO, E., ISOLA, M. & TEBALDI, G. 2011. The role of fillers on cracking behavior of mastics and asphalt mixtures. *Journal of the Association of Asphalt Paving Technologists,* 80**,** 161-192.

MUTHEN, K. M. 1998. Foamed Asphalt Mixes - Mix Design Procedure. CSIR TRANSPORTEK South Africa.

NEWCOMB, D. 2005. Warm Mix Asphalt: Wave of the Future. Lanham, Maryland.: National Asphalt Pavement Association.

NEWCOMB, D. E., ARAMBULA, E., YIN, F., ZHANG, J., BHASIN, A., LI, W. & AREGA, Z. 2015. Properties of foamed asphalt for WMA applications. National cooperative highway research program.

OKE, O. L. 2010. *A study on the development of guidelines for the production of bitumen emulsion stabilised RAPs for roads in the tropics.* PhD, The University of Nottingham.

P. J. RUCKEL, L. L. KOLE, F. ABEL, R. E. ZATOR, J. W. BUTTON & E. J.A. 1980. Foamix asphalt advances Scherocman.

PELLINEN, T. K. & WITCZAK, M. W. 2002. Stress Dependent Master Curve Construction for Dynamic (Complex) Modulus. . *Journal of the Association of Asphalt Paving Technologists,* Vol. 71, **,** pp. 281–309.

PELLINEN, T. K., WITCZAK, M. W. & BONAQUIST, R. F. 2002. Asphalt Mix Master Curve Construction using Sigmoidal Fitting Function with Non-Linear Least Squares Optimization Technique. . *Proceedings of 15th ASCE Engineering Mechanics Conference June 2-5, 2002, Columbia University, New York.*

PERKINS, S. W. 2009. Synthesis of warm mix asphalt paving strategies for use in Montana highway construction.

PQ CORPORATION 2012. Production, testing and compaction details. Advera WMA. Warm mix asphalt.

PROWELL, B. D., HURLEY, G. C. & FRANK, B. 2012. Warm-Mix Asphalt: Best Practice. Lanham, USA: National Asphalt Pavement Association (NAPA).

REYHANEH RAHBAR-RASTEGAR, DANIEL, J. S. & REINKE, G. 2017. Comparison of asphalt binder and mixture cracking parameters. *Road Materials and Pavement Design***,** 2164-7402.

ROBERTS, F. L., KANDHAL, P. S., BROWN, E. R., LEE, D.-Y. & KENNEDY, T. W. 1996. Hot mix asphalt materials, mixture design and construction.

ROWE, G. 2011. Evaluation of the relationship between asphalt binder properties and non-load related cracking. *Journal of the Association of Asphalt Paving Technologists,* 80**,** 649-662.

ROWE, G., BAUMGARDNER, G. & SHARROCK, M. 2009. Functional Forms for Master Curve Analysis of Bituminous Materials. *Proceedings of 7th International RILEM 229 Symposium ATCBM09 on Advanced Testing and Characterization of Bituminous Materials,* 1**,** pp. 81–91

SARGAND, S. M., FIGUEROA, J. L., EDWARDS, W. & AL-RAWASHDEH, A. S. 2009. Performance Assessment of Warm Mix Asphalt (WMA) Pavements Athens, Ohio Ohio University.

SCHOLZ, T. V. 1995. *Durability of bituminous paving mixtures.* The University of Nottingham.

SHAH, A., MCDANIEL, R., HUBER, G. & GALLIVAN, V. 2007. Investigation of Properties of Plant-Produced Reclaimed Asphalt Pavement Mixtures. *Transportation Research Record: Journal of the Transportation Research Board,* 1998**,** 103-111.

SMITH, B. & HESP, S. 2000. Crack pinning in asphalt mastic and concrete. *Transportation Research Record: Journal of the Transportation Research Board,* 1728**,** 75-81.

SONDAG, M., CHADBOURN, B. & DRESCHER, A. 2002. Investigation of Recycled Asphalt Pavement (RAP) Mixtures. St. Paul, Minnesota.

STUART, K. D. 1990. Moisture damage in highway mixtures – a state-of the -art report. FHWA-RA-90-019. . Washington, DC: Department of Transportation.

SUNARJONO, S. 2008. *The influence of foamed bitumen characteristics on cold-mix asphalt properties.* The University of Nottingham.

TABAKOVI´C, A., GIBNEY, A., MCNALLY, C. & GILCHRIST, M. D. 2010. Influence of recycled asphalt pavement on fatigue performance of asphalt concrete base courses. . *Journal of Materials in Civil Engineering* 22**,** 643-650.

THOM, N. 2014. *Principles of Pavement Engineering,* University of Nottingham, Nottingham, UK, ICE Publishing.

TONG, Y., LUO, R. & LYTTON, R. L. 2013. Modeling water vapor diffusion in pavement and its influence on fatigue crack growth of fine aggregate mixture. *Transportation Research Record: Journal of the Transportation Research Board,* 2373**,** 71-80.

TRAXLER, R. 1963. Durability of asphalt cements. *Association of Asphalt Paving Technologist,,* 32**,** pp. 44-63.

VASCONCELOS, K. L. 2010. *Moisture diffusion in asphalt binders and fine aggregate mixtures.* PhD thesis, Texas A&M University.

W. S. MOGAWER, A. J. AUSTERMAN, KASSEM, E. & E. MASAD 2010. Moisture Damage Characteristics of Warm Mix Asphalt Mixtures. *Journal of the Association of Asphalt Paving Technologists,* 80**,** pp 491-526.

WEST, R., WILLIS, J. R. & MARASTEANU, M. 2013. Improved Mix Design, Evaluation, and Materials Management Practices for Hot Mix Asphalt with High Reclaimed Asphalt Pavement Content. *NCHRP Report 752*. Transportation Research Board.

WIRTGEN 2010. Wirtgen Cold Recycling Technology. 3rd edition ed. Windhagen, Germany.

XIAO, F., JORDAN, J. & AMIRKHANIAN, S. 2009a. Laboratory investigation of moisture damage in warm-mix asphalt containing moist aggregate. *Journal of the Transportation Research Board,* 14**,** 115-124.

YUSSOF, N. I. M. 2012. *Modelling the linear viscoelastic rheological properties of bituminous binders.* The University of Nottingham.

ZEIADA, W. A., ALOSSTA, A. A. & KALOUSH, K. E. 2017. Laboratory evaluation of foaming-based and chemical-based warm mix asphalt. *Innovative Infrastructure Solutions*.

Appendix A

DMA testing protocol for determining the rheological properties in FAM materials

Testing protocol for

Determining the rheological properties of FAM materials using the Kinexus Dynamic Shear Rheometer

1. SCOPE

This test method covers the determination of the linear viscoelastic properties (i.e. dynamic shear modulus, $|G^*|$ and phase angle, δ) of Fine aggregate matrix (FAM) materials when tested in dynamic shear using solid fixture configuration.

2. DEFINITIONS

2.1 Dynamic Shear modulus, $|G^*|$ can be defined as the absolute magnitude of the peak shear stress, σ, divided by the absolute magnitude of the peak shear strain, γ.

2.2 Phase angle, δ, corresponds to the angle in degrees between a sinusoidal applied strain and the resultant stress, in a controlled strain testing mode, or between a sinusoidal applied stress and the resultant strain, in a controlled stress testing mode.

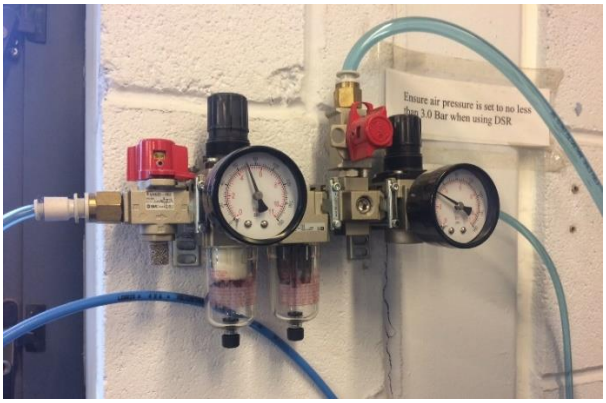
2.3 Fine aggregate matrix (FAM), is a combination of bitumen with the finest portion of the aggregates (e.g. particles size smaller than 1 mm) and air voids, which are present in the full scale asphalt mixtures.

3. APPARATUS

The kinexus DSR type machine is illustrated in the figure below. The DSR test system for FAM materials consists of an upper and lower geometry, an environmental controller cartridge, a loading unit, and a data acquisition system. The figure below illustrates the testing configuration in the rheometer.

4. PREPARATION OF APPARATUS

4.1 Verify the air supply is on, and ensure that the air pressure is set to no less than 3.0 bar.

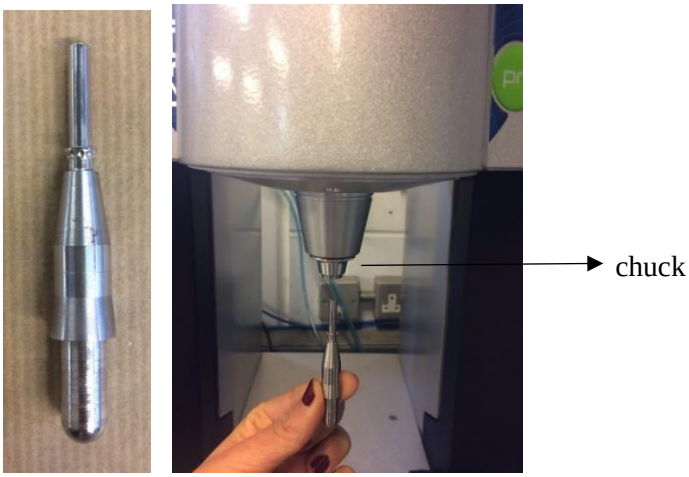


4.2 Turn the machine on by pressing the button that is on the back of the rheometer, at the low right corner.

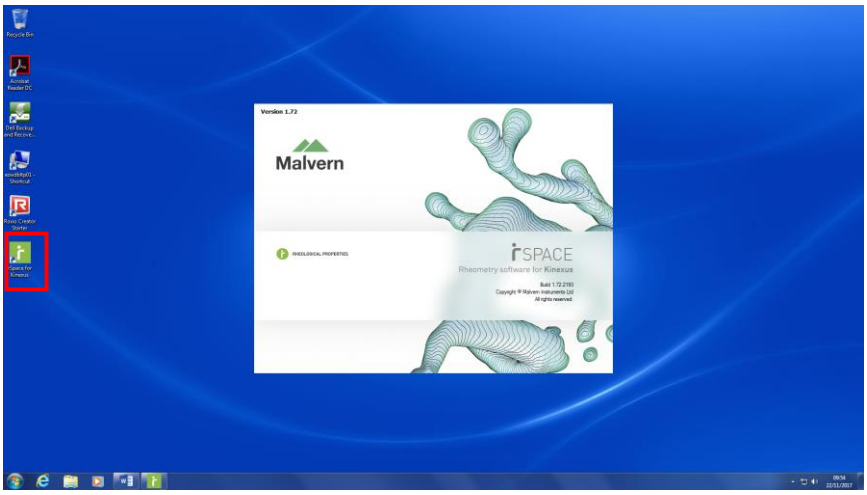
4.3 Turn on the computer.

4.4 Push the cartridge gently into the slot. Move the lever on the cartridge fully to the right to lock.

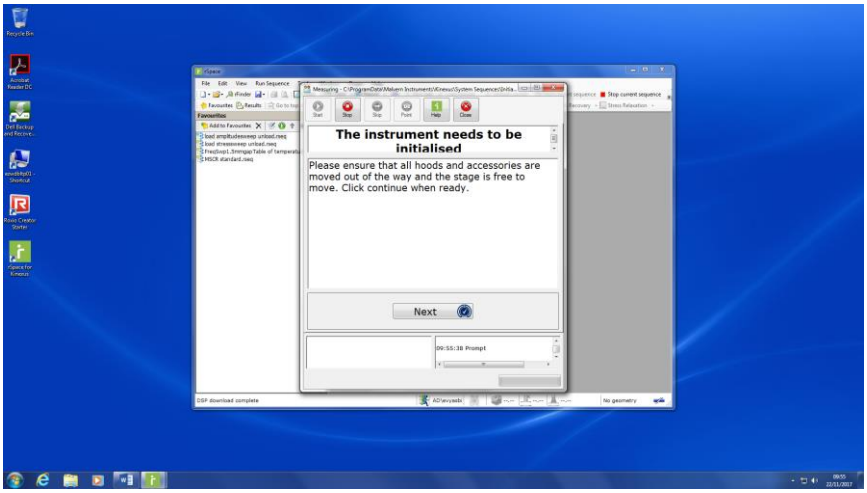
4.5 Take the spindle and push it gently into the chuck as shown in the figure below.



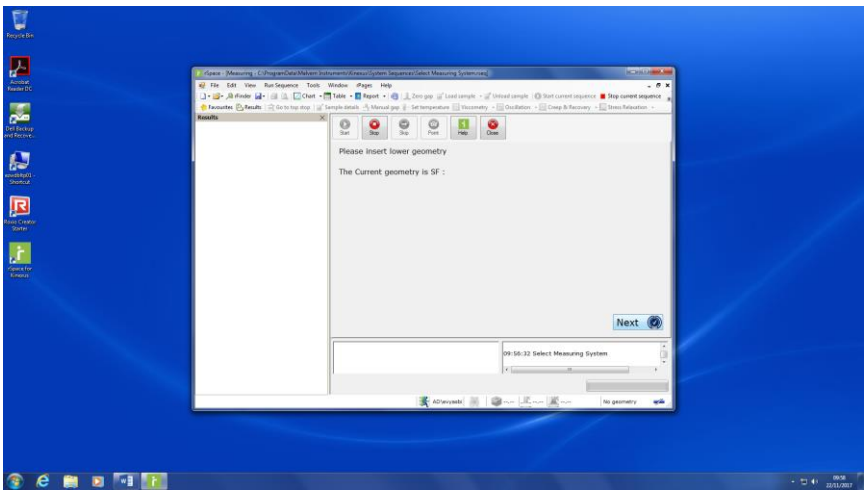
4.6 Double click the kinexus software icon on the desktop, to open the software.



4.7 The software will initialise, and then click on the “Next” bottom.



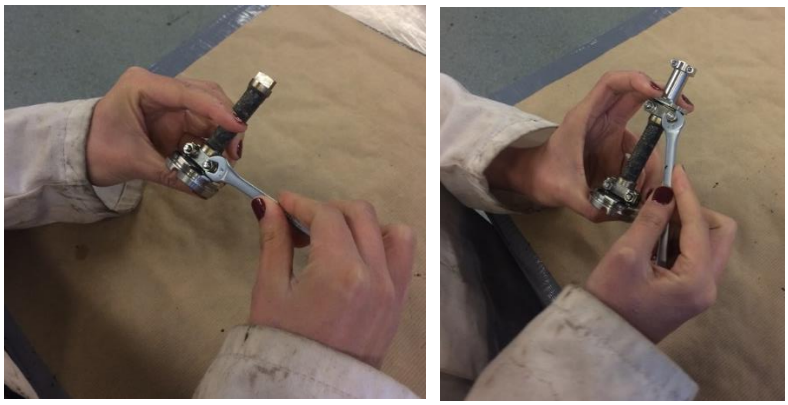
4.8 The system will automatically recognise the solid fixture configuration. Click the "Next" bottom to continue.

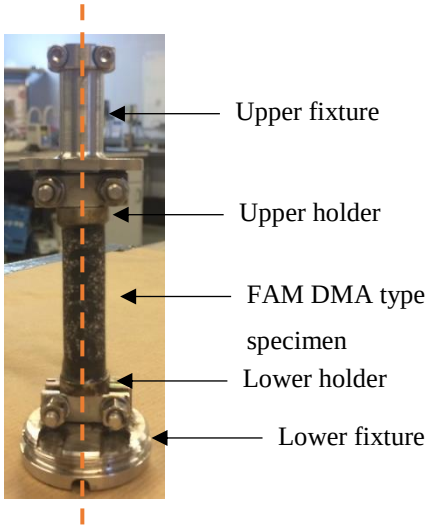


4.9 The next step is to perform the zero gap. Place a sample cover on the top of the torsion cup as shown in the figure below. Ensure that the rheometer is clear. The head of the machine will go to the lowest position until the upper geometry reaches the sample cover, and then return to its initial position automatically.

5. PREPARATION OF SAMPLES AND MOUNTING

5.1 Attach the upper and lower fixtures to the sample using a screw as shown in the figures below. The sample should be correctly aligned, as shown in the figure below.



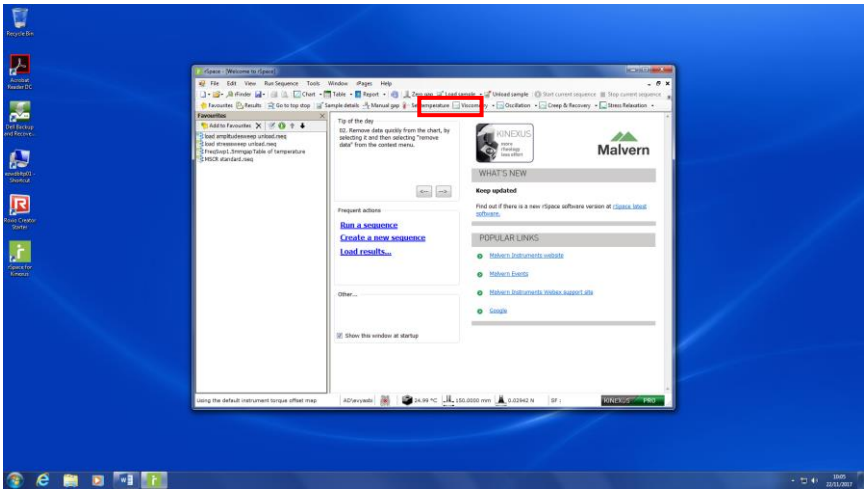


Correctly aligned

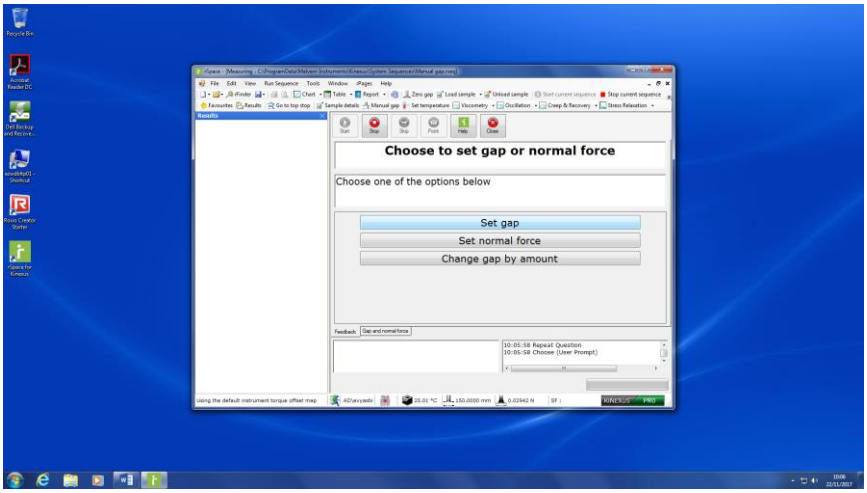
5.2 Insert the sample in the cylindrical base as shown in the figures below.

5.3 Place the cylinder in the hole in the cartridge and move the lever to the right to secure.

5.4 Click on the “Manual gap” bottom, to set the required gap.



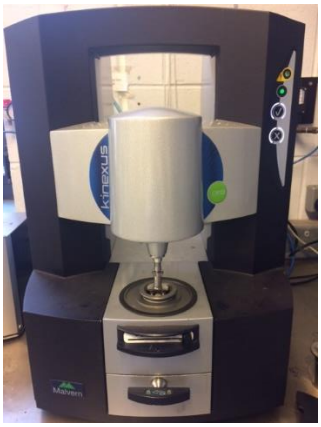
5.5 Click on the “Set gap” bottom.



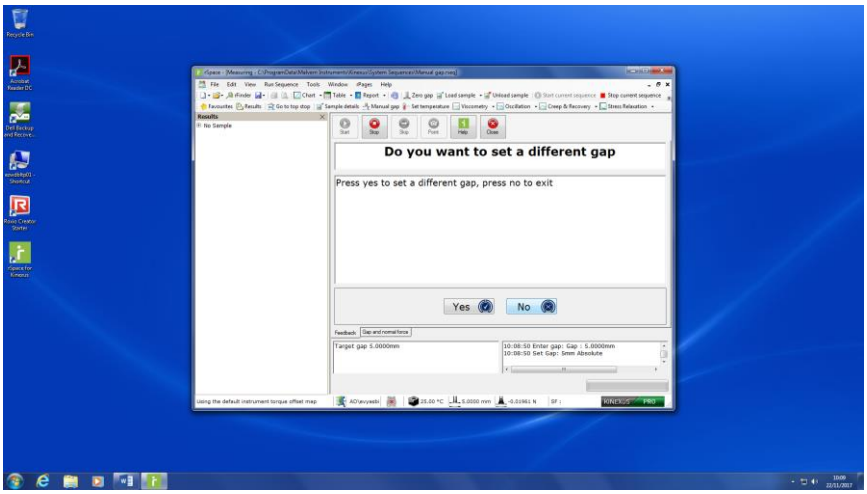
5.6 Customise the gap size by entering the value on the displayed window, until the upper geometry fits in the upper fixture.

Upper geometry

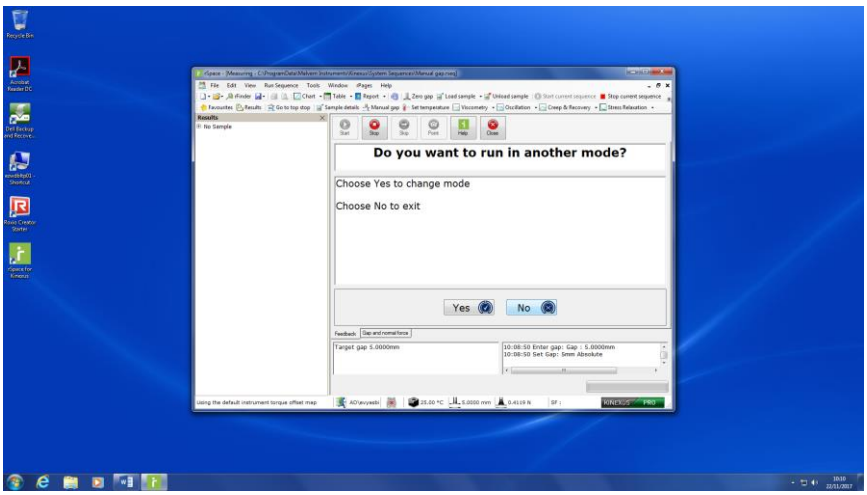
Upper fixture



5.7 When the gap size is set, click on the "No" bottom to continue.



5.8 Then again click on the "No" bottom to continue.

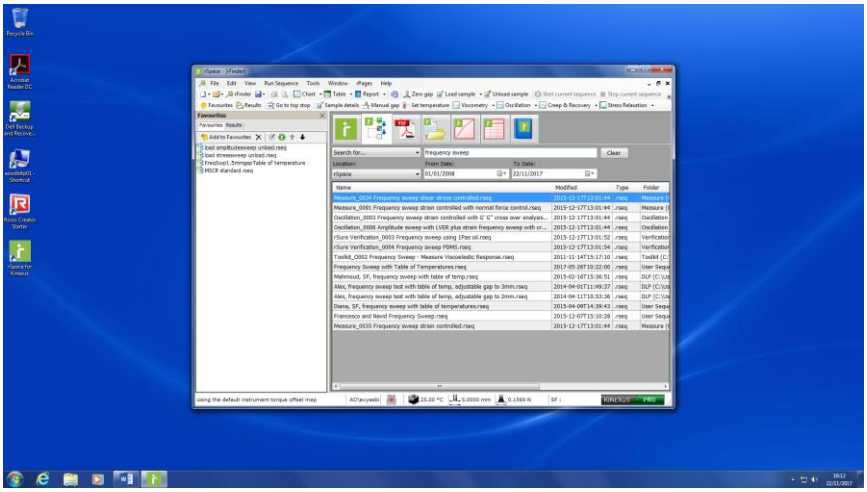


5.9 Screw the upper geometry and place the sample covers as shown in the figures below.

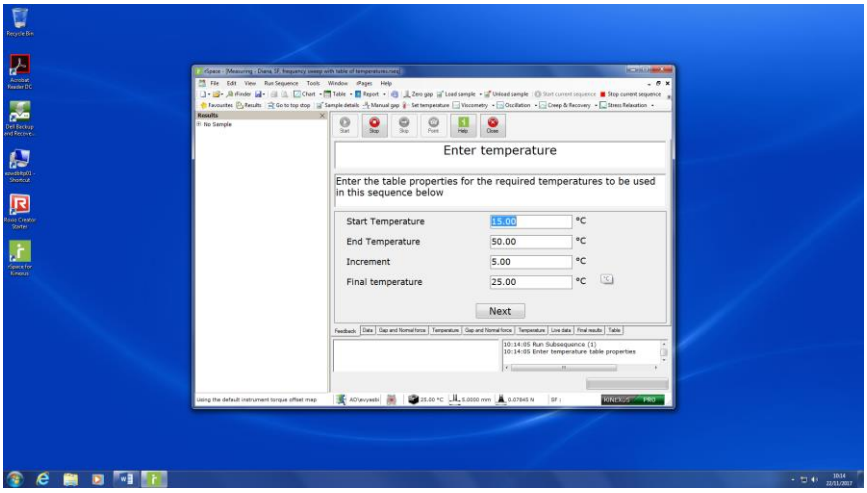


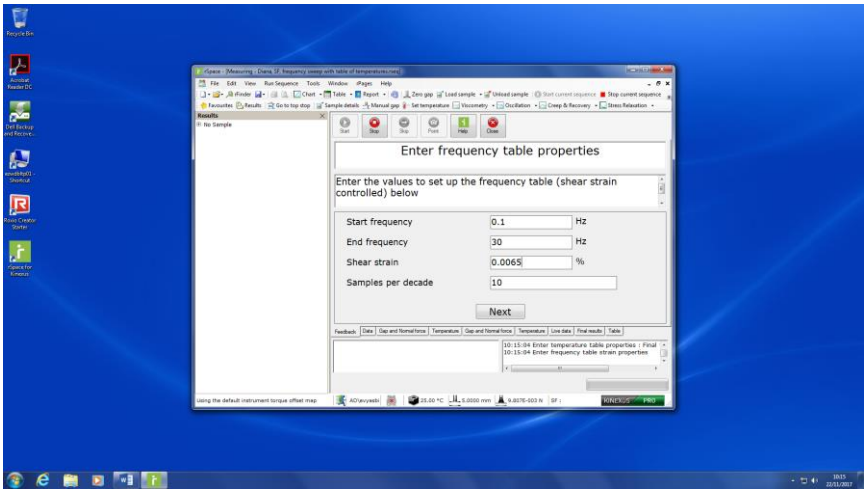
6. TESTING PROCEDURE FOR FREQUENCY AND TEMPERATURE SWEEP

6.1 Click on the rFinder bottom to find the appropriate sequence. The kynexus contains default sequences. Type “frequency sweep” to find a list of related sequences. For running a sequence in FAM materials, the geometry in the sequence must be set up for a solid configuration. Double-click on an item in the list to run the sequence or open the file.

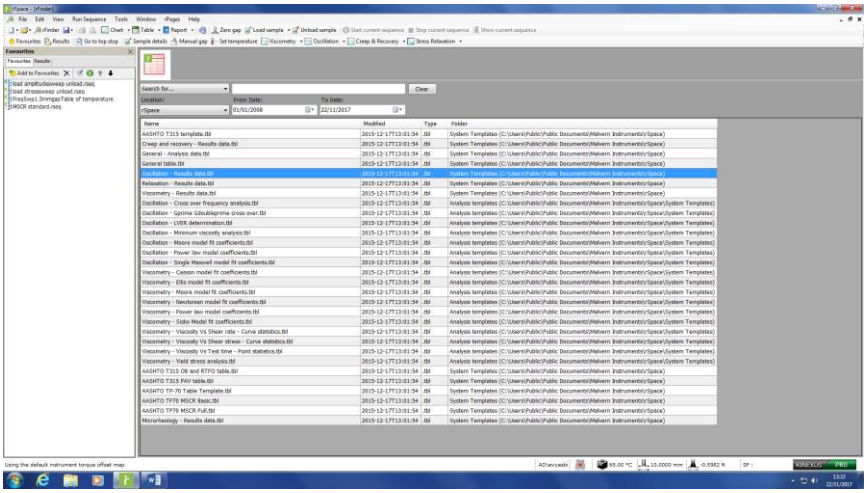


6.2 Enter the test conditions (i.e. temperature and frequency ranges, and shear strain).

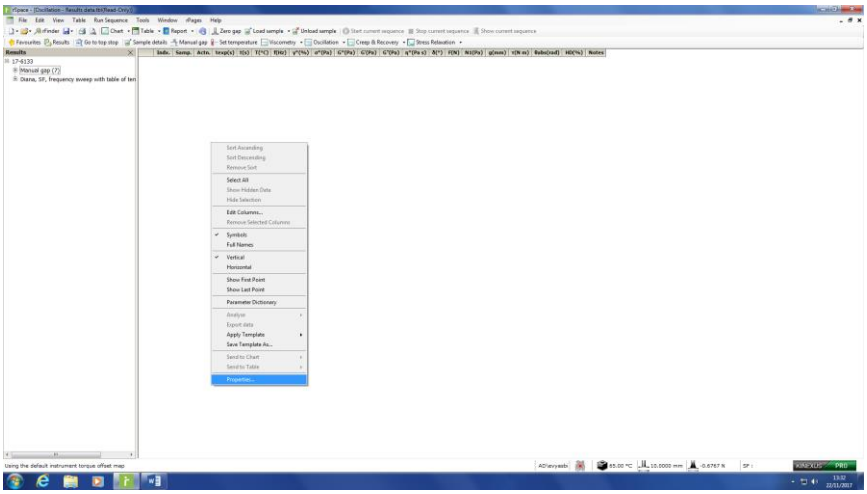




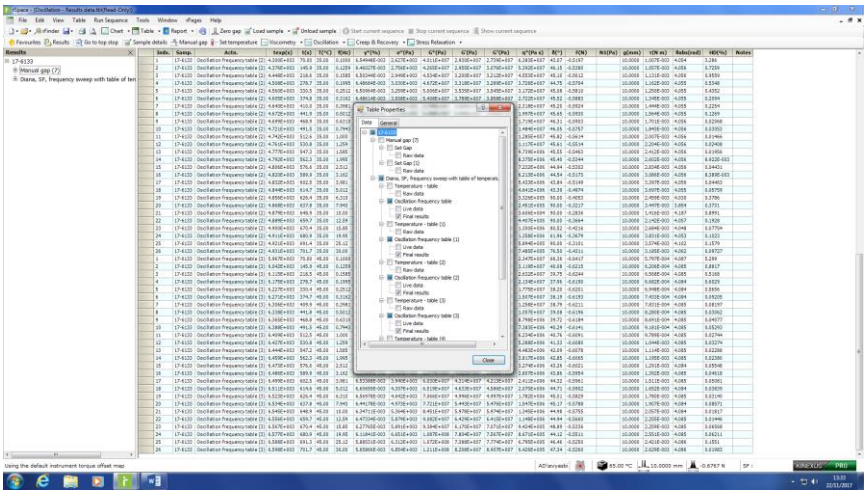
6.3 Once the test is over, to save the data click on the "Table" button and double click on "Oscillation – Results data" on the options.



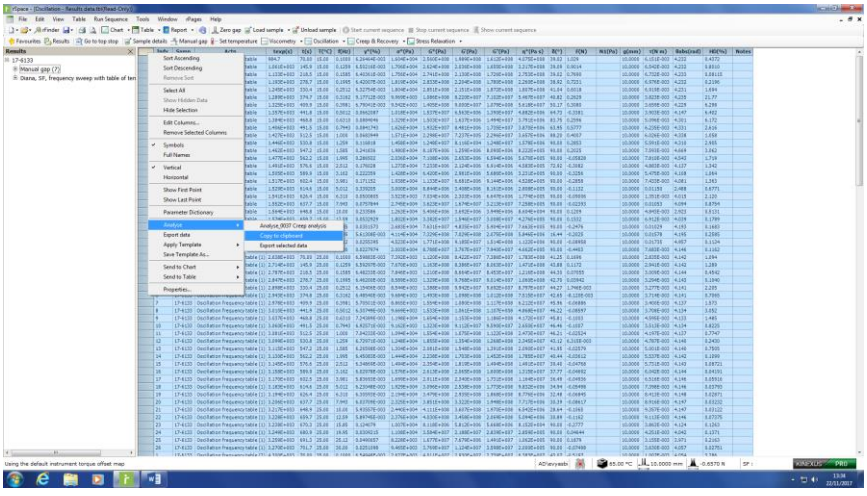
6.4 Right click on the screen and select “Properties”



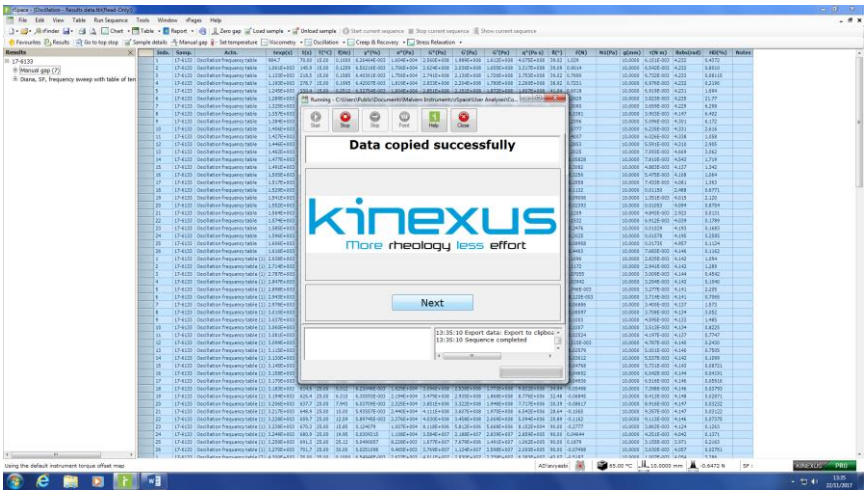
6.5 Select the “Final data” for all the tested temperatures.



6.6 Go to the top left of the table to select all data.



6.7 Click on “Next”, and then open an Excel file and paste the data.



Appendix B

Detailed procedure for the Statistical Analysis

Procedure for the Statistical analysis (T-test) for $|G^*|$ and δ

In this section, the procedure followed to conduct the statistical analysis (t-test) on the dynamic shear modulus and phase angle of the mixtures is explained. In general, the analysis was conducted comparing the $|G^*|$ and δ values obtained from the sweep frequency and temperature tests, at all testing temperatures and loading frequencies, between mixtures.

The results were plotted in the form of *p*-value as a function of the loading frequency, at all the tested temperatures. The red dashed line in each figure represents the borderline of statistical significance, where results yielding a *p*-value equal to or lower than 0.05 are statistical significant. Otherwise, results yielding a *p*-value above 0.05 are considered not statistical significant.

Example Statistical Analysis for Chapter 5: In this Chapter the corresponding $|G^*|$ values obtained from the sweep frequency and temperature tests, at all testing temperatures and loading frequencies, for all the mixtures containing RAP (i.e. Mechanical foamed-RAP, Zeolite-FAM, HMA-RAP and 100%RAP mixtures) were compared to those of the reference Virgin HMA to evaluate significance and in this way assess level of blending between RAP and virgin binders (Figure B1). In this analysis, the null hypothesis is that the means of the $|G^*|$ values of the corresponding RAP with reference to the Virgin HMA mixture are not significantly different. As an example, the $|G^*|$ values of two mixtures (e.g. 100% RAP and Zeolite-RAP-120C) in Figure B1, are compared to those of the reference Virgin HMA.

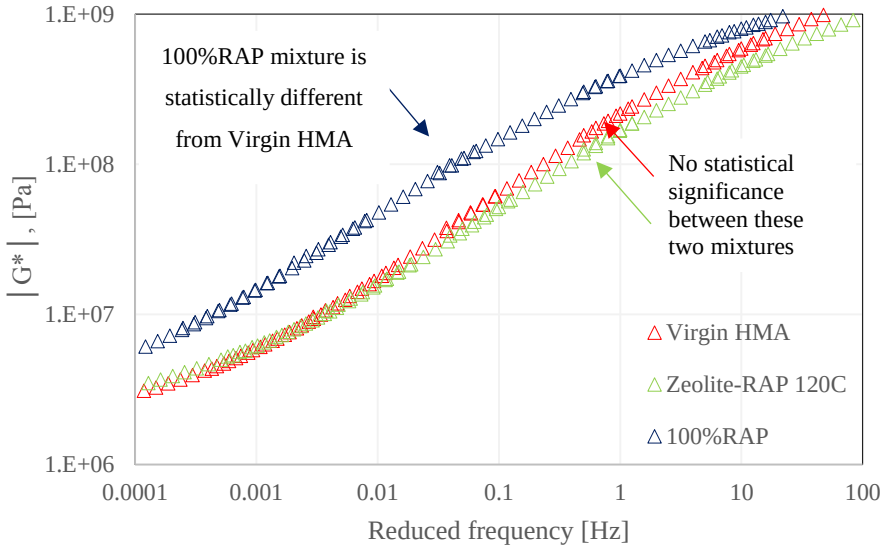


Figure B1. $|G^*|$ master curve for Zeolite-RAP, 100%RAP and Virgin HMA mixtures

Figure B2 a, shows the results of comparing the $|G^*|$ values of the 100%RAP mixture with those of the Virgin HMA. In this case, all the p-values are below 0.05 indicating the $|G^*|$ values between

these mixtures are statistically significant and therefore, these mixtures can be considered different. Figure B2 b shows the results of comparing the Zeolite-RAP-120C mixture with the Virgin HMA. In this case, in general the p-values exhibit values above 0.05, indicating that the $|G^*|$ values of these mixtures are not statistical significant. In other words, there is no statistical evidence that these values are different. This means in this case, that the properties (i.e. $|G^*|$ values) of the Zeolite-RAP-120C mixture are comparable to those of the Virgin HMA, which implies that the incorporation of RAP material and mixing at 120ºC generated a mixture with comparable properties to those of the Virgin HMA, as explained in Chapter 5. From this figure, it is also noticeable that some p-values of the $|G^*|$ values measured at 15 and 25ºC (red and orange curves respectively) are below 0.05. Thus indicating that the statistical significance also varies with temperature and frequency. These values correspond to the high-frequency end in the master curve (Figure B1), where visible differences can be observed.

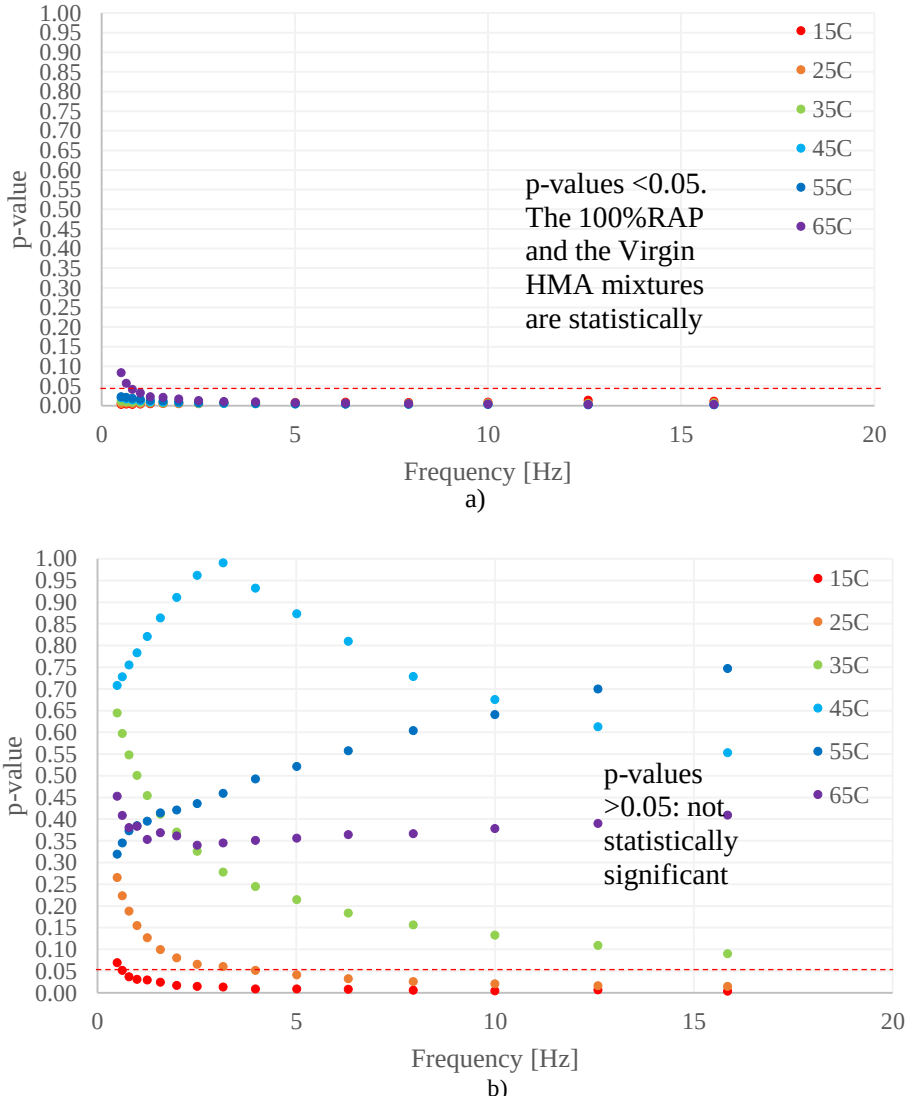


Figure B2. Statistical analysis comparing Virgin HMA with a) 100%RAP and b) Zeolite-FAM 120C

Example Statistical Analysis for Chapter 6: In this Chapter, the same procedure was followed to evaluate in this case, the statistical significance of the $|G^*|$ and δ values of the mixtures after been subjected to the environmental conditioning protocol, for all conditioning times, with respect to their initial (i.e. dry condition). The following figures present an example for the Virgin HMA.

The $|G^*|$ and δ master curves are initially presented in Figure B3 and Figure B4 respectively. The statistical analysis was conducted by comparing the $|G^*|$ raw data from the tested samples for each conditioning time (i.e. 6, 15 and 30 days), to those of the initial condition, for each evaluated mixture. The null hypothesis is that the $|G^*|$ values after 6 days conditioning, are not significantly different from the $|G^*|$ values at the initial condition.

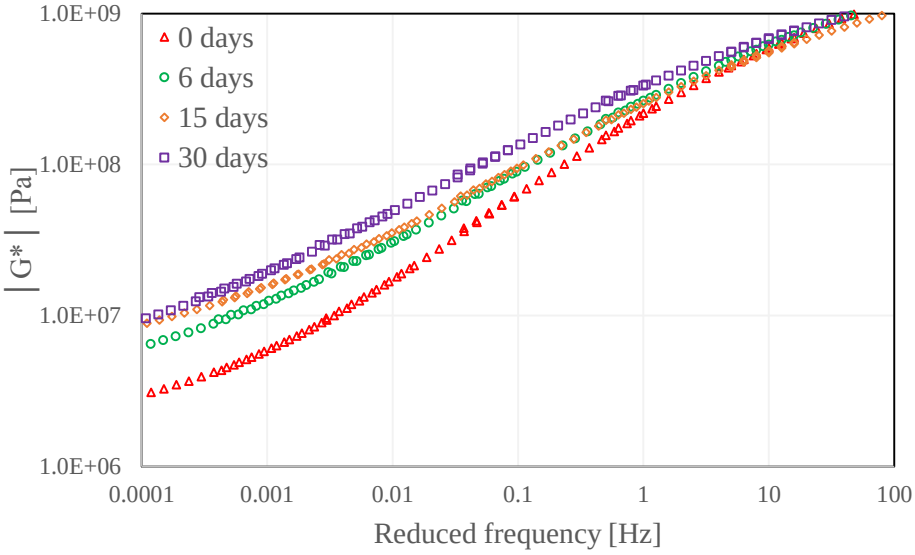


Figure B3. Master curve of |G*| for the Virgin HMA

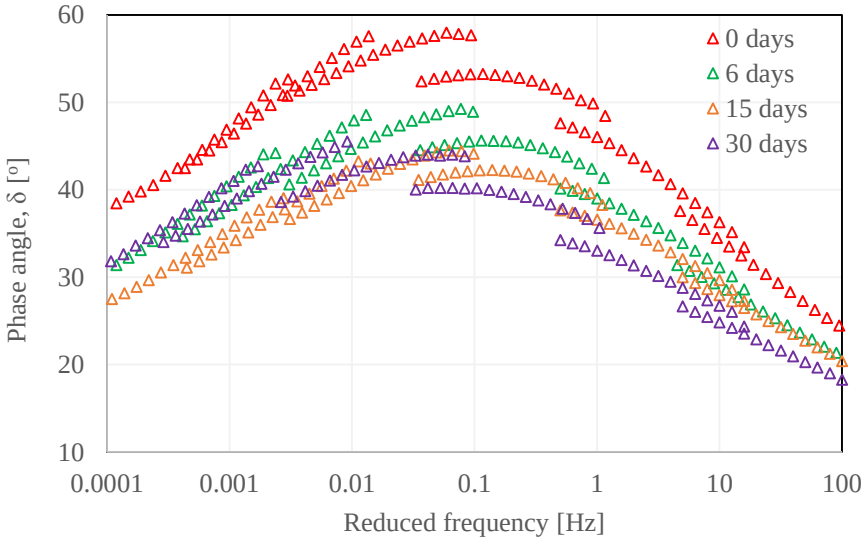


Figure B4. Master curve of phase angle for the Virgin HMA

In terms of the $|G^*|$ (see figures below), the statistical analysis shows that in general most of the *p*-values are below 0.05, thus indicating that the $|G^*|$ values after all conditioning times (e.g. 6, 15 and 30 days) are statistically different from the initial condition. This is seen in the $|G^*|$ master curve, where the $|G^*|$ values are increasing progressively with conditioning time, which reflects that the properties of the material start changing after 6 days conditioning in this case for this analysis. Some *p*-values are above 0.05, for instance at low testing temperatures of 15 and 25ºC, which correspond to the high-frequency end in the $|G^*|$ master curve. Thus the statistical significance varies with the testing frequency and temperature. The same procedure was applied to the δ values, where in this case, the *p*-values are almost below or within the statistical significance limit for all frequencies and temperatures, for all conditioning times (see figures below), meaning that there is a statistical difference between the materials evaluated.

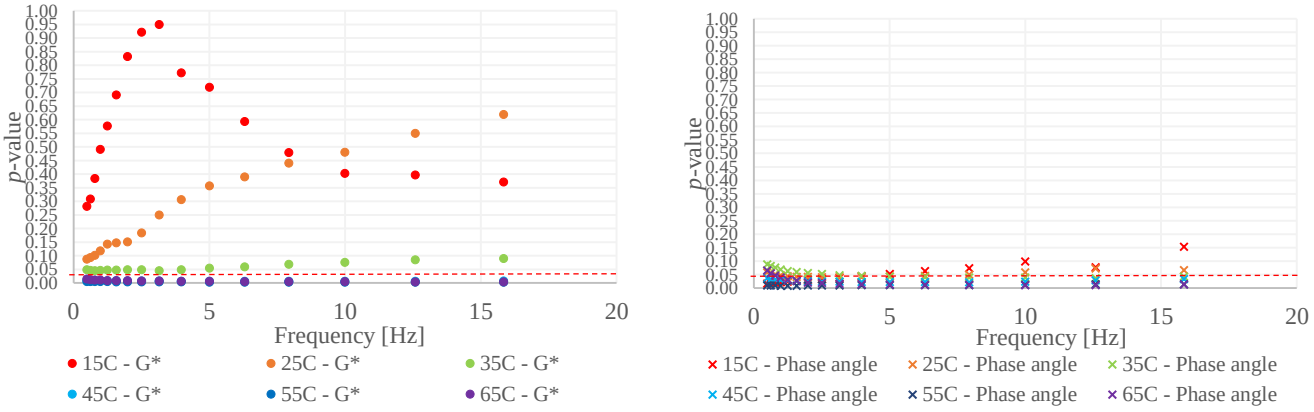


Figure B5. Virgin HMA 0 – 6 days a) $|G^*|$ and, b) δ

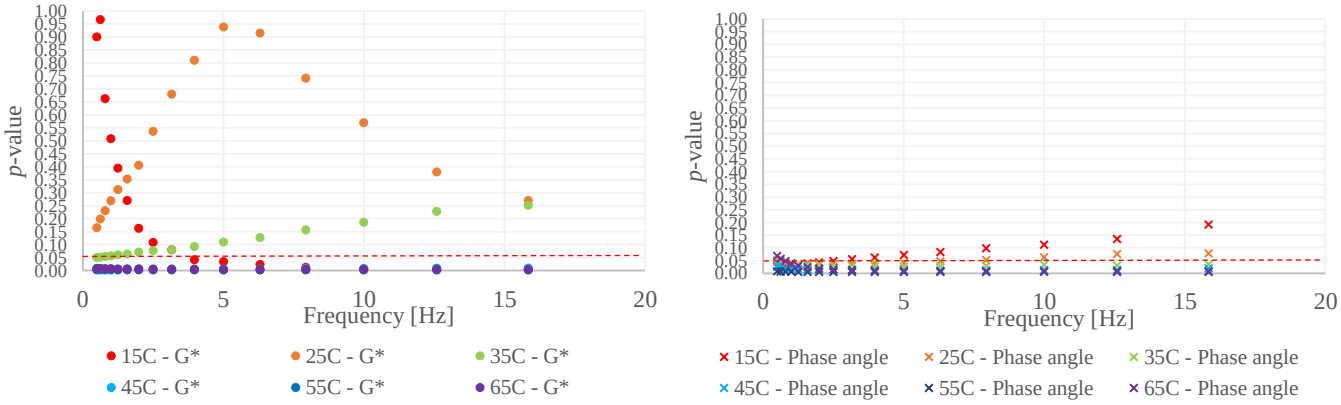


Figure B6. Virgin HMA 0 – 15 days a) $|G^*|$ and, b) δ

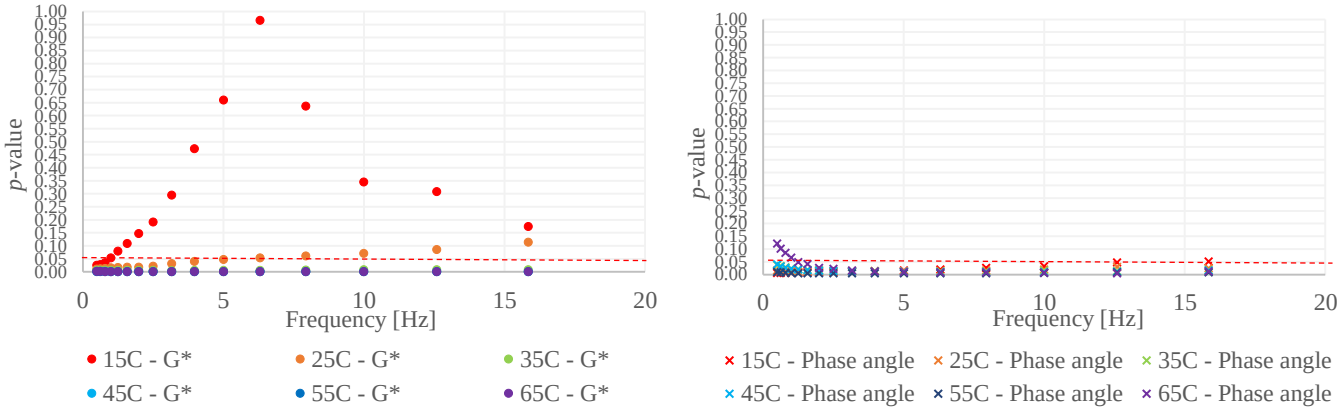


Figure B7. Virgin HMA 0 – 30 days a) $|G^*|$ and, b) δ

Appendix C

Phase angle master curves for the environmental conditioning in Chapter 6

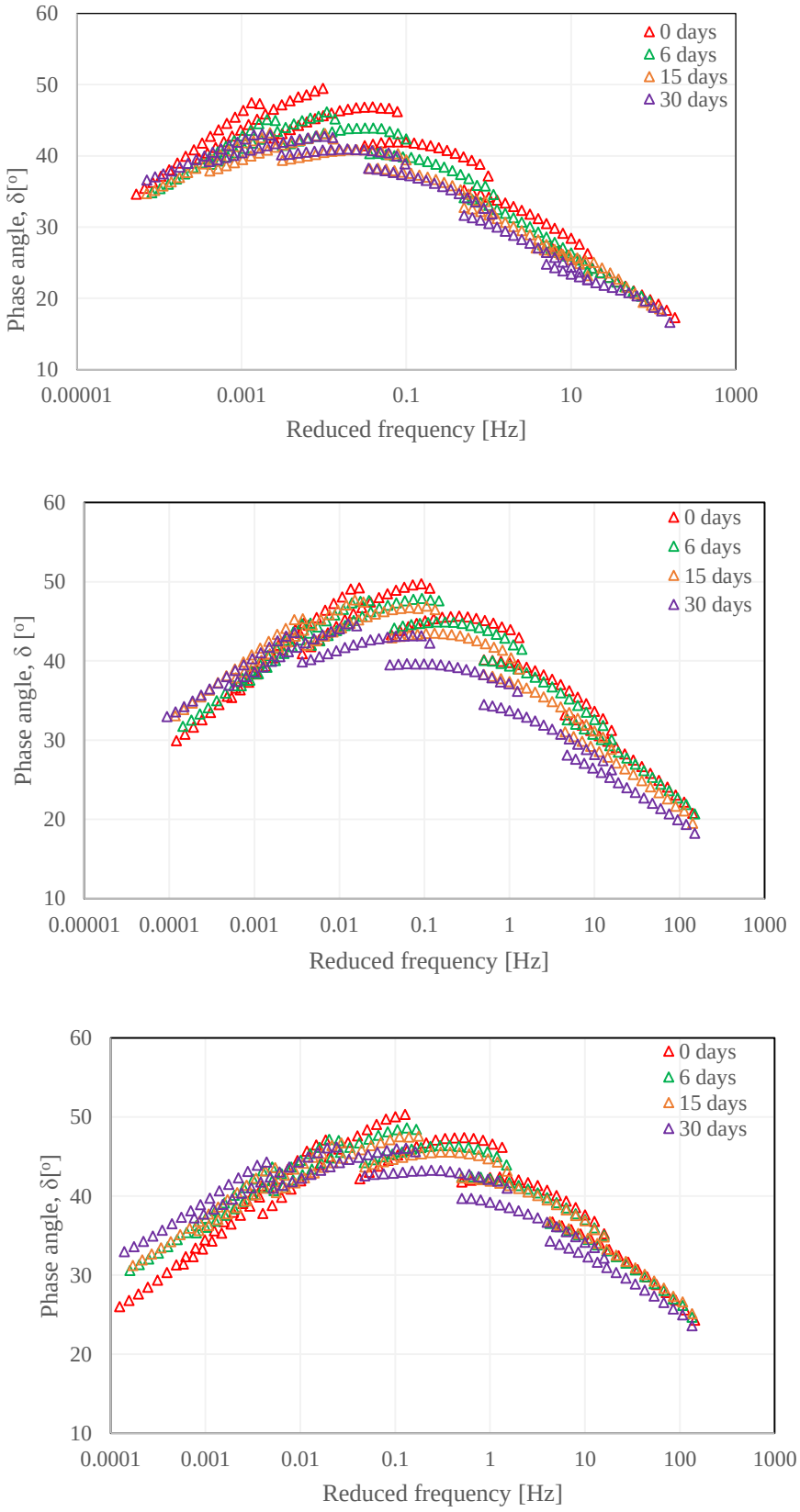
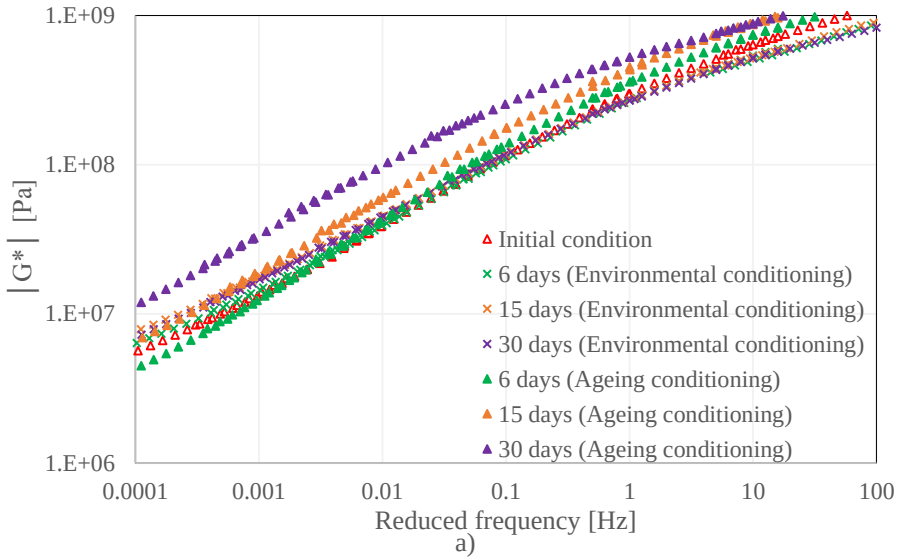


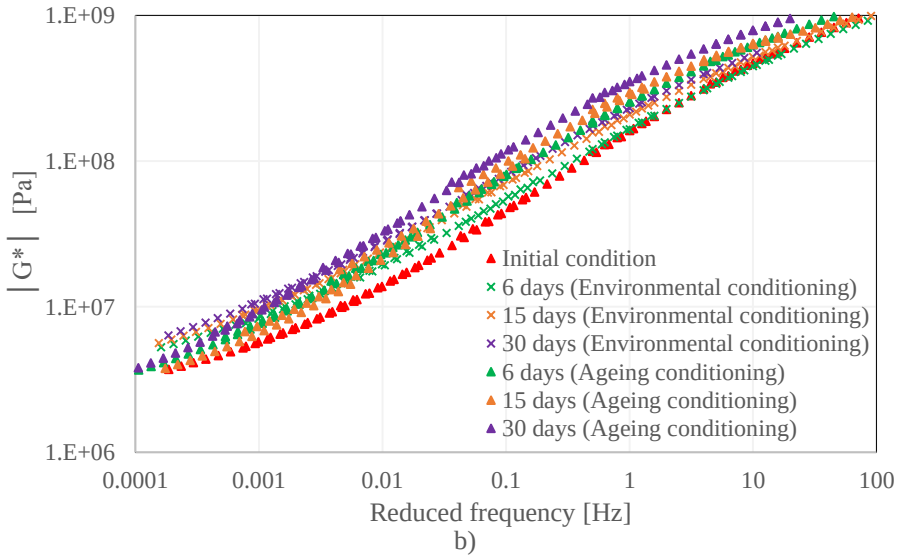
Figure C1. Master curve of δ at a reference temperature of 25°C for the a) HMA-RAP, b) Mechanical foamed-RAP at 120°C, and c) Zeolite-RAP at 120°C.

Appendix D

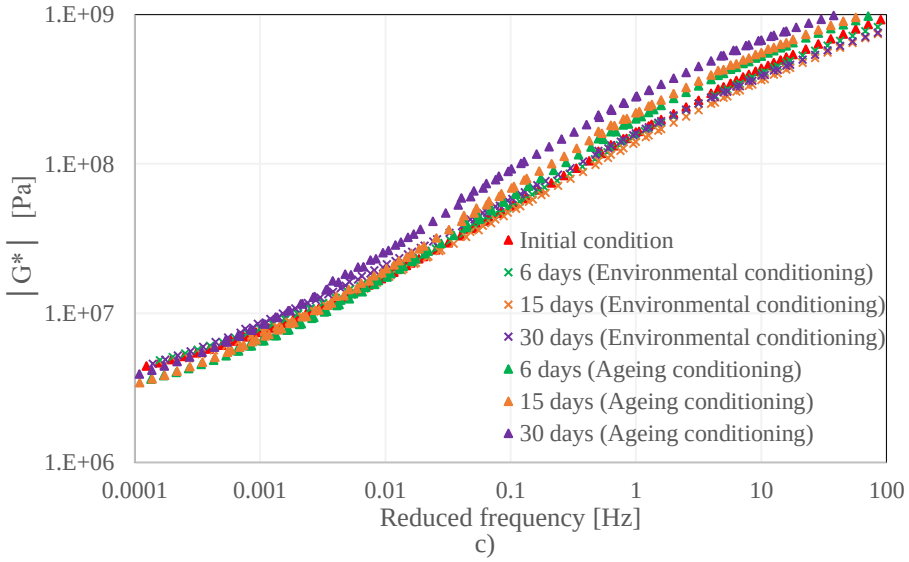
$|G^*|$ master curves for combined effect of laboratory ageing and moisture effects in Chapter 6



a)



b)



c)

Figure D1. Master curve of $|G^*|$ at a reference temperature of 25°C for the a) HMA-RAP, b) Mechanical foamed-RAP at 90ºC, and c) Zeolite-RAP at 120ºC.

Appendix E

DSR data for recovered binders before and after SATS conditioning in Chapter 7

Table E1. Complex modulus (Pa) - Binder recovered from Virgin HMA mixture (Initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	7383000	3231000	1185000	393500	122400	35750	11160	3742	1403	559	262	123	64	33	21
0.16 Hz	9643000	4309000	1630000	558000	178100	52620	17090	6116	2374	898	407	190	101	51	31
0.25 Hz	12300000	5710000	2223000	783800	256000	77760	25740	9440	3598	1406	648	300	162	81	51
0.40 Hz	15030000	7242000	2973000	1090000	368100	114600	38550	14320	5559	2181	1014	473	254	130	80
0.63 Hz	18740000	9343000	3977000	1508000	522400	167400	57470	21560	8538	3416	1590	753	405	205	129
1.00 Hz	23100000	11870000	5249000	2066000	737800	243200	85230	32410	13210	5305	2471	1179	634	326	203
1.59 Hz	28570000	15020000	6840000	2804000	1033000	351400	125800	48280	20110	8172	3856	1853	1002	516	326
2.51 Hz	34200000	18660000	8853000	3772000	1436000	503600	184700	71510	30520	12480	5974	2903	1573	817	501
3.98 Hz	40950000	23040000	11330000	5009000	1975000	713000	270000	106500	46040	19120	9187	4503	2454	1293	812
6.31 Hz	48550000	28310000	14370000	6586000	2699000	1002000	389800	157400	68230	28990	14080	6983	3837	2017	1268
10.00 Hz	57150000	34180000	17990000	8574000	3644000	1396000	557300	229100	100600	43460	21420	10720	5899	3126	1980

Table E2. Phase angle (º) - Binder recovered from Virgin HMA mixture (Initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	51	57	63	69	74	78	80	83	81	89	88	86	88	90	89
0.16 Hz	47	54	61	67	72	76	80	83	84	86	88	88	88	88	89
0.25 Hz	46	52	59	65	71	75	79	82	84	86	87	88	89	89	88
0.40 Hz	44	50	57	64	69	74	78	81	83	85	87	88	88	89	89
0.63 Hz	42	48	55	62	68	73	77	80	82	85	86	87	88	88	88
1.00 Hz	40	46	53	60	67	72	76	79	81	84	86	87	88	88	88
1.59 Hz	39	45	52	59	65	70	75	78	79	83	85	86	87	88	88
2.51 Hz	36	43	50	57	64	69	74	77	78	83	84	86	87	86	88
3.98 Hz	35	41	48	55	62	68	72	75	76	82	84	85	86	87	88
6.31 Hz	34	40	46	54	61	67	71	74	74	81	83	85	86	86	89
10.00 Hz	33	39	45	52	59	65	70	72	70	80	83	84	86	85	89

Table E3. Complex modulus (Pa) - Binder recovered from Virgin HMA mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	26680000	13820000	6783000	2928000	1120000	408700	156700	60420	26570	5648	2206	950	427	218	110
0.16 Hz	24970000	18340000	8506000	3834000	1504000	567100	220300	87770	39180	8456	3419	1468	662	344	174
0.25 Hz	37930000	20160000	10360000	4991000	2012000	774600	309100	125900	56620	12570	5211	2248	1037	537	274
0.40 Hz	43940000	23940000	12530000	6195000	2626000	1055000	432400	180100	82560	18620	7807	3430	1598	838	430
0.63 Hz	51410000	28510000	14870000	7849000	3450000	1423000	603900	257200	120000	27410	11780	5227	2474	1302	677
1.00 Hz	56490000	33580000	17520000	9777000	4479000	1907000	832000	362300	172100	39930	17440	7904	3795	2006	1047
1.59 Hz	68640000	39650000	21120000	11880000	5764000	2535000	1133000	506200	245900	58000	25850	11890	5774	3090	1626
2.51 Hz	77300000	46340000	25210000	14150000	7340000	3345000	1535000	702100	348900	83310	38040	17770	8722	4724	2513
3.98 Hz	87060000	53110000	29740000	16840000	9310000	4376000	2062000	967600	491700	118500	55200	26280	13070	7213	3849
6.31 Hz	96630000	60400000	34960000	19490000	11700000	5683000	2746000	1324000	682300	168600	79800	38390	19480	10880	5888
10.00 Hz	106400000	67900000	40650000	23150000	14490000	7317000	3631000	1792000	941100	236100	114700	55950	28770	16310	8882

Table E4. Phase angle (º) - Binder recovered from Virgin HMA mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	38	42	48	54	60	65	69	73	77	83	85	85	85	86	87
0.16 Hz	16	49	46	51	57	62	67	71	75	78	81	84	85	86	87
0.25 Hz	33	38	44	49	55	61	66	70	74	77	80	83	84	86	87
0.40 Hz	31	37	42	48	54	60	64	68	72	75	79	82	84	85	86
0.63 Hz	30	36	41	47	53	58	63	67	71	74	78	81	83	84	86
1.00 Hz	31	34	40	45	51	57	62	66	70	73	76	79	82	84	85
1.59 Hz	28	33	38	44	50	55	60	65	68	71	75	78	81	83	85
2.51 Hz	27	32	37	43	49	54	59	63	67	70	74	77	80	82	84
3.98 Hz	26	31	36	41	47	53	58	62	66	69	73	76	79	81	83
6.31 Hz	25	29	35	40	46	52	57	61	66	67	71	75	78	80	83
10.00 Hz	24	29	34	39	45	51	56	60	65	66	70	74	77	80	82

Table E5. Complex modulus (Pa) - Binder recovered from HMA-RAP mixture (initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	13890000	7713000	3829000	1705000	702000	281300	111700	43950	18930	8105	3511	1555	697	330	164
0.16 Hz	15580000	9455000	4840000	2205000	921900	377700	153600	61730	27070	12220	5293	2366	1074	515	255
0.25 Hz	20080000	11450000	6040000	2795000	1194000	500600	208200	85700	38110	17200	7765	3517	1649	797	404
0.40 Hz	23420000	13790000	7225000	3477000	1540000	662300	280900	118600	53590	24690	11250	5217	2477	1224	621
0.63 Hz	27540000	16550000	8876000	4375000	1983000	873400	378500	162600	74740	35170	16430	7736	3724	1879	964
1.00 Hz	32380000	19730000	10810000	5456000	2534000	1143000	505900	222100	103800	49800	23660	11380	5564	2848	1477
1.59 Hz	37440000	23620000	13080000	6750000	3216000	1487000	673200	301900	143700	70240	33850	16600	8245	4291	2250
2.51 Hz	43710000	27620000	15800000	8331000	4051000	1925000	890100	407300	197100	97190	48020	23980	12110	6400	3415
3.98 Hz	50320000	32440000	18960000	10210000	5087000	2474000	1165000	545000	268900	134700	67600	34540	17670	9455	5119
6.31 Hz	57940000	37770000	22630000	12460000	6352000	3167000	1518000	726500	363000	186600	94900	49010	25490	13920	7631
10.00 Hz	65910000	43910000	26800000	15100000	7878000	4023000	1963000	960200	484300	253100	131800	69320	36560	20220	11270

Table E6. Phase angle (º) - Binder recovered from HMA-RAP mixture (initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	37	41	45	50	54	59	63	67	71	75	78	82	84	85	86
0.16 Hz	40	39	43	47	52	56	61	65	69	72	76	79	82	84	85
0.25 Hz	34	38	41	46	51	55	60	64	67	71	75	78	80	83	84
0.40 Hz	33	36	40	45	50	54	58	62	66	70	73	77	80	82	84
0.63 Hz	32	35	40	44	49	53	57	61	65	68	72	75	78	81	83
1.00 Hz	31	35	39	43	48	52	56	60	64	67	71	74	77	80	82
1.59 Hz	30	33	38	42	47	51	55	59	63	65	69	73	75	78	81
2.51 Hz	29	32	37	41	46	50	54	58	62	64	68	71	74	77	80
3.98 Hz	28	32	36	40	45	49	54	57	61	63	67	70	73	76	78
6.31 Hz	27	31	35	39	44	49	53	56	61	61	66	69	72	75	77
10.00 Hz	27	30	35	39	43	48	52	56	60	61	65	68	71	74	76

Table E7. Complex modulus (Pa) - Binder recovered from HMA-RAP mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	20530000	12190000	6897000	3470000	1581000	685200	293600	124400	52460	25940	11230	4875	2149	949	469
0.16 Hz	22400000	13980000	8247000	3610000	2220000	875100	398300	169900	72880	38500	16180	7487	3258	1462	728
0.25 Hz	28120000	17350000	10080000	5294000	2495000	1137000	514100	226300	99870	51230	23270	10550	4838	2215	1117
0.40 Hz	31380000	19600000	11780000	6328000	3134000	1461000	675000	303200	137500	72340	32560	15250	7127	3326	1701
0.63 Hz	36660000	23220000	14140000	7769000	3885000	1858000	878700	404300	186400	98940	46690	22040	10500	4991	2602
1.00 Hz	41940000	26780000	16640000	9367000	4798000	2349000	1135000	533800	251000	135900	65140	31420	15280	7394	3916
1.59 Hz	46980000	31070000	19650000	11190000	5884000	2945000	1453000	701500	336100	184000	90170	44450	22040	10870	5855
2.51 Hz	51550000	34460000	22790000	13410000	7189000	3677000	1861000	916600	448300	246200	124000	62510	31620	15800	8678
3.98 Hz	60650000	40390000	26550000	15880000	8688000	4581000	2362000	1185000	594700	329500	170000	86940	44860	22870	12770
6.31 Hz	66950000	46420000	30620000	18750000	10510000	5642000	2976000	1530000	784300	434200	229000	120300	63090	32850	18600
10.00 Hz	73990000	52280000	35320000	22050000	12620000	6924000	3730000	1958000	1022000	564900	307300	165300	88240	46680	26810

Table E8. Phase angle (º) - Binder recovered from HMA-RAP mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	31	35	39	43	48	53	57	61	65	69	73	77	81	83	84
0.16 Hz	35	38	44	34	52	50	54	59	63	72	71	74	78	81	83
0.25 Hz	28	31	35	39	44	49	53	58	62	65	69	73	77	80	82
0.40 Hz	25	29	33	37	43	47	52	56	60	64	68	72	75	79	81
0.63 Hz	26	29	33	37	42	46	51	55	59	62	66	70	74	77	80
1.00 Hz	25	29	32	36	41	45	49	54	58	60	65	68	72	76	79
1.59 Hz	25	28	31	35	40	44	48	53	57	58	63	67	71	74	77
2.51 Hz	26	28	30	34	39	43	47	51	55	57	62	66	69	73	76
3.98 Hz	23	26	30	34	38	42	46	51	55	55	60	65	68	71	75
6.31 Hz	24	26	29	33	37	41	46	50	53	53	59	63	67	70	73
10.00 Hz	22	26	29	32	37	41	45	49	53	51	57	62	66	69	72

Table E9. Complex modulus (Pa) - Binder recovered from Zeolite-RAP-120C mixture (initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	6913000	3249000	1374000	528000	185600	64970	22910	9159	3615	1250	515	261	127	62	36
0.16 Hz	8876000	4216000	1830000	721000	259000	93920	34350	14210	5562	1943	807	408	200	99	57
0.25 Hz	11140000	5441000	2397000	971600	359700	134500	49770	20690	8352	2994	1265	639	319	155	92
0.40 Hz	13340000	6713000	3081000	1303000	497000	190900	72990	30830	12640	4591	1964	998	501	246	144
0.63 Hz	16420000	8464000	3989000	1735000	684400	270600	106400	44360	18990	7009	3050	1558	786	390	228
1.00 Hz	19970000	10560000	5115000	2288000	934600	381100	152900	63880	28190	10630	4682	2413	1228	608	357
1.59 Hz	24260000	13090000	6500000	2996000	1262000	530100	219700	92890	41440	15970	7172	3726	1911	957	566
2.51 Hz	28800000	15990000	8203000	3887000	1688000	731200	312200	135500	61070	23910	10880	5725	2968	1497	904
3.98 Hz	34290000	19470000	10250000	5011000	2245000	1002000	438100	195400	88520	35500	16470	8718	4563	2327	1386
6.31 Hz	40150000	23420000	12710000	6391000	2960000	1354000	613700	279200	128800	52060	24680	13250	6987	3585	2165
10.00 Hz	46580000	27880000	15600000	8100000	3872000	1825000	844000	392700	182400	76220	36570	19950	10620	5506	3313

Table E10. Phase angle (º) - Binder recovered from Zeolite-RAP-120C mixture (initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	47	52	57	63	67	71	76	79	81	86	87	87	88	88	87
0.16 Hz	44	49	54	60	65	70	74	78	81	84	86	87	88	88	88
0.25 Hz	42	47	53	59	64	69	73	76	80	83	85	86	87	88	87
0.40 Hz	41	46	51	57	62	67	71	75	78	82	84	86	87	88	88
0.63 Hz	39	44	50	56	61	66	70	74	77	81	83	85	86	87	88
1.00 Hz	38	43	48	54	60	65	69	73	76	80	83	84	86	87	88
1.59 Hz	36	41	47	53	58	63	68	72	75	79	81	84	85	86	87
2.51 Hz	35	40	46	51	57	62	66	71	74	78	80	83	84	85	87
3.98 Hz	34	39	44	50	56	61	65	70	73	77	79	82	84	85	86
6.31 Hz	33	38	43	49	54	60	64	69	72	76	79	81	83	84	86
10.00 Hz	32	37	42	47	53	59	63	68	72	75	78	80	82	83	86

Table E11. Complex modulus (Pa) - Binder recovered from Zeolite-RAP-120C mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	7718000	3589000	1680000	729500	283100	102200	39540	16760	6664	3877	1568	660	321	155	78
0.16 Hz	9808000	4204000	2057000	939000	382700	142100	56360	24360	9904	6010	2403	1030	502	242	121
0.25 Hz	11010000	5086000	2530000	1188000	508300	195200	79430	34990	14620	8731	3636	1589	781	382	193
0.40 Hz	12850000	6131000	3040000	1490000	675800	268000	111600	50250	21380	12900	5518	2437	1202	597	306
0.63 Hz	14970000	7364000	3728000	1869000	888400	364200	155600	71570	31030	19230	8317	3723	1865	928	481
1.00 Hz	17060000	8757000	4531000	2325000	1158000	492000	215200	101300	44680	28220	12420	5662	2858	1438	744
1.59 Hz	19400000	10360000	5460000	2873000	1490000	659300	295600	142500	63930	41320	18490	8510	4377	2217	1155
2.51 Hz	22130000	12190000	6493000	3495000	1877000	876600	403400	198600	90990	59820	27180	12740	6621	3419	1778
3.98 Hz	24920000	14270000	7716000	4251000	2332000	1151000	545900	275400	129200	85500	39870	18960	9939	5213	2744
6.31 Hz	27890000	16490000	9120000	5142000	2860000	1498000	732300	379300	181500	121900	57710	27950	14890	7925	4191
10.00 Hz	30710000	18770000	10700000	6108000	3503000	1935000	973300	515700	252300	172100	83360	40830	22030	11900	6364

Table E12. Phase angle (º) - Binder recovered from Zeolite-RAP-120C mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	39	45	50	56	61	66	70	74	77	81	83	85	86	87	88
0.16 Hz	46	42	47	53	58	63	68	72	75	78	82	84	86	87	86
0.25 Hz	35	41	46	52	57	62	67	71	74	78	81	83	85	86	87
0.40 Hz	34	39	44	50	56	61	65	69	73	76	80	82	84	85	86
0.63 Hz	33	38	43	49	54	59	64	68	72	75	78	81	83	85	86
1.00 Hz	32	36	42	47	53	58	63	67	70	74	77	80	82	84	85
1.59 Hz	31	35	41	46	51	57	61	65	69	72	76	79	81	83	85
2.51 Hz	30	34	40	45	50	55	60	64	68	71	75	78	80	82	84
3.98 Hz	29	34	39	44	49	54	59	63	66	70	74	77	79	82	83
6.31 Hz	28	33	38	43	48	53	58	62	64	69	72	76	78	81	82
10.00 Hz	28	32	37	42	47	52	57	61	62	67	72	75	78	80	82

Table E13. Complex modulus (Pa) - Binder recovered from Mechanical foamed-RAP-90C mixture (initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	8818000	4129000	1685000	609300	199400	62540	21750	8115	3203	1353	566	268	132	67	38
0.16 Hz	11340000	5381000	2255000	832700	282100	90100	32230	12280	4958	2093	886	427	211	108	60
0.25 Hz	14040000	6929000	2958000	1126000	393100	129100	47490	18340	7568	3212	1384	671	335	170	97
0.40 Hz	16890000	8497000	3812000	1514000	545400	184200	69320	27200	11480	4929	2150	1052	525	270	151
0.63 Hz	20640000	10660000	4948000	2032000	753500	261700	100500	40170	17290	7538	3321	1641	825	424	239
1.00 Hz	24950000	13260000	6343000	2696000	1032000	369200	144800	59010	25870	11410	5097	2538	1291	671	379
1.59 Hz	30280000	16300000	8067000	3544000	1401000	516900	207200	86170	38230	17140	7780	3919	2007	1058	591
2.51 Hz	35720000	20060000	10170000	4620000	1889000	717600	294300	125100	55960	25710	11780	6025	3104	1654	931
3.98 Hz	42190000	24410000	12720000	5974000	2524000	991700	415400	179800	82720	38160	17820	9199	4772	2581	1453
6.31 Hz	49420000	29460000	15780000	7669000	3347000	1358000	580900	256100	119400	56200	26630	13930	7305	3972	2254
10.00 Hz	57790000	35210000	19430000	9743000	4398000	1839000	806700	360000	168100	82600	39510	20930	11110	6072	3470

Table E14. Phase angle (º) - Binder recovered from Mechanical foamed-RAP-90C mixture (initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	46	51	57	63	68	73	77	80	83	86	87	88	88	89	89
0.16 Hz	42	48	54	60	66	71	75	79	82	84	86	87	88	88	86
0.25 Hz	41	46	53	59	65	70	74	78	80	83	85	86	87	88	89
0.40 Hz	40	45	51	57	63	68	73	76	79	82	84	86	87	88	89
0.63 Hz	38	44	50	56	62	67	71	75	78	81	83	85	86	87	88
1.00 Hz	36	42	48	54	61	66	70	74	77	80	82	84	86	87	88
1.59 Hz	35	41	47	53	59	65	69	73	76	79	81	83	85	86	87
2.51 Hz	34	39	45	51	58	63	68	72	75	78	80	83	84	85	87
3.98 Hz	33	38	44	50	56	62	67	71	75	77	79	82	84	85	86
6.31 Hz	32	37	43	49	55	61	66	70	74	76	79	81	83	84	85
10.00 Hz	31	36	41	48	54	60	64	69	74	75	78	80	82	84	85

Table E15. Complex modulus (Pa) - Binder recovered from Mechanical foamed-RAP-90C mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	13500000	7506000	3725000	1684000	645000	230200	83720	33280	11660	4761	1949	806	393	182	93
0.16 Hz	13440000	7733000	4437000	2192000	887100	321900	119000	48200	17300	7172	3006	1245	614	288	147
0.25 Hz	19750000	11440000	5967000	2816000	1150000	435000	166300	69550	25420	10700	4548	1938	954	452	234
0.40 Hz	22770000	13580000	7300000	3558000	1499000	587200	231500	99650	37380	15940	6856	2953	1480	705	367
0.63 Hz	27190000	16620000	9122000	4574000	1983000	796800	322100	141500	53800	23450	10300	4515	2291	1100	576
1.00 Hz	31880000	19880000	11170000	5767000	2575000	1068000	442400	199400	77440	34230	15320	6838	3527	1696	903
1.59 Hz	36570000	23330000	13510000	7180000	3315000	1421000	603100	278200	110800	49590	22610	10300	5406	2618	1399
2.51 Hz	42970000	27590000	16430000	8926000	4234000	1880000	817300	386500	157700	71680	33400	15340	8202	4021	2162
3.98 Hz	48880000	32460000	19680000	10990000	5385000	2462000	1099000	532700	221800	102200	48790	22790	12330	6119	3334
6.31 Hz	55520000	37940000	23440000	13420000	6777000	3192000	1466000	726100	312200	145500	70070	33510	18380	9258	5081
10.00 Hz	63150000	43950000	27700000	16270000	8463000	4108000	1941000	979500	430300	204100	100500	48780	27130	13880	7732

Table E16. Phase angle (º) - Binder recovered from Mechanical foamed-RAP-90C mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	38	42	48	53	59	64	69	73	77	80	84	85	86	87	86
0.16 Hz	27	31	49	50	56	61	66	71	75	78	81	84	86	87	87
0.25 Hz	34	38	43	49	55	60	65	70	74	78	81	83	85	86	86
0.40 Hz	32	36	41	47	53	59	64	68	72	76	79	82	84	86	86
0.63 Hz	32	36	41	46	52	57	63	67	71	75	78	81	83	85	86
1.00 Hz	30	35	39	44	50	56	61	66	70	73	77	80	82	84	85
1.59 Hz	29	33	38	43	49	55	60	65	68	72	75	78	81	83	85
2.51 Hz	28	32	37	42	47	53	59	63	67	70	74	77	80	82	84
3.98 Hz	28	31	35	40	46	52	57	62	66	69	73	76	79	81	83
6.31 Hz	27	30	35	39	45	51	56	61	64	68	72	75	78	80	82
10.00 Hz	25	29	34	39	44	50	55	60	62	66	71	74	77	80	82

Table E17. Complex modulus (Pa) - Binder recovered from Mechanical foamed-RAP-120C mixture (initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	9411000	4850000	2096000	799700	285300	97880	36350	13150	5011	2244	962	428	205	106	58
0.16 Hz	12470000	6212000	2748000	1076000	391600	137400	52700	19590	7491	3434	1484	670	326	161	88
0.25 Hz	14010000	7809000	3555000	1433000	532400	192200	75330	28880	11200	5197	2287	1038	516	253	136
0.40 Hz	17100000	9429000	4463000	1867000	721800	266900	107300	42440	16610	7846	3497	1610	802	401	214
0.63 Hz	20650000	11670000	5673000	2452000	975100	369900	151800	61740	24450	11810	5347	2486	1252	627	341
1.00 Hz	24730000	14270000	7138000	3183000	1307000	509100	213700	88590	35730	17540	8084	3809	1937	976	530
1.59 Hz	29310000	17310000	8906000	4103000	1733000	695700	298900	125900	52020	25940	12140	5815	2984	1520	835
2.51 Hz	34680000	20950000	11050000	5237000	2282000	944700	415600	177400	74900	38050	18110	8804	4580	2354	1305
3.98 Hz	40580000	25070000	13610000	6647000	2983000	1273000	573400	248300	108200	55440	26810	13180	6941	3608	2033
6.31 Hz	47440000	29770000	16640000	8373000	3875000	1705000	783500	345800	155300	80220	39440	19740	10530	5507	3130
10.00 Hz	54560000	35210000	20200000	10460000	4991000	2268000	1060000	476800	218400	115200	57540	29180	15770	8338	4781

Table E18. Phase angle (º) - Binder recovered from Mechanical foamed-RAP-120C mixture (initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	43	47	53	59	64	68	72	77	80	83	85	86	87	88	89
0.16 Hz	41	44	50	56	61	66	71	74	78	81	84	86	87	87	88
0.25 Hz	39	43	48	54	60	65	69	73	77	81	83	85	86	87	89
0.40 Hz	37	42	47	53	59	64	68	72	76	79	82	84	86	87	87
0.63 Hz	36	40	46	52	57	63	67	71	74	78	81	83	85	86	87
1.00 Hz	35	39	44	50	56	61	66	70	73	76	80	82	84	85	86
1.59 Hz	33	38	43	49	55	60	65	69	72	75	78	81	83	85	86
2.51 Hz	32	37	42	48	53	59	64	68	70	74	77	80	82	84	85
3.98 Hz	31	36	41	46	52	58	63	67	69	73	76	79	81	83	84
6.31 Hz	30	35	40	45	51	57	62	65	67	72	75	78	80	82	83
10.00 Hz	29	34	39	44	50	56	61	65	66	71	74	77	79	81	83

Table E19. Complex modulus (Pa) - Binder recovered from Mechanical foamed-RAP-120C mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	16270000	8875000	4291000	1833000	717600	271600	106800	44880	16900	7470	3082	1329	581	280	141
0.16 Hz	16310000	9545000	4505000	2353000	956700	377100	149000	64170	24860	11430	4703	2040	902	435	215
0.25 Hz	22680000	12940000	6625000	2987000	1245000	518600	208300	90330	35730	16370	7016	3093	1393	676	333
0.40 Hz	25790000	15100000	8009000	3733000	1626000	672100	282100	127100	51190	23710	10420	4683	2127	1046	524
0.63 Hz	30760000	18240000	9900000	4748000	2102000	895700	385300	178000	73070	34720	15460	7059	3268	1616	808
1.00 Hz	35550000	21600000	12000000	5929000	2703000	1186000	523000	246900	103500	49990	22720	10570	4954	2482	1245
1.59 Hz	40660000	25720000	14510000	7332000	3443000	1560000	704000	340500	145300	71450	33110	15630	7464	3786	1922
2.51 Hz	46590000	29790000	17190000	9026000	4373000	2031000	941700	464600	203100	101400	47930	23100	11150	5751	2954
3.98 Hz	53270000	34520000	20430000	10990000	5477000	2623000	1251000	632400	282000	142800	68710	33780	16520	8640	4494
6.31 Hz	59900000	39970000	24210000	13370000	6836000	3368000	1649000	849000	390100	202400	97960	48900	24300	12920	6805
10.00 Hz	67620000	45760000	28360000	16080000	8477000	4292000	2154000	1127000	528400	276200	138500	70320	35430	19100	10210

Table E20. Phase angle (º) - Binder recovered from Mechanical foamed-RAP-120C mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	34	40	45	51	56	62	66	70	74	78	81	84	84	85	86
0.16 Hz	28	37	36	48	54	59	64	68	72	75	79	82	83	85	86
0.25 Hz	31	36	41	47	53	57	62	67	71	75	78	81	83	84	85
0.40 Hz	29	34	39	45	51	56	61	65	69	73	77	80	82	84	85
0.63 Hz	29	34	39	44	49	55	60	64	68	71	75	78	81	83	84
1.00 Hz	28	32	37	43	48	54	59	63	67	70	74	77	80	82	84
1.59 Hz	27	31	36	41	47	52	57	62	65	69	73	76	79	81	83
2.51 Hz	25	31	35	40	46	51	56	60	64	67	71	75	77	80	82
3.98 Hz	26	29	34	39	44	50	55	59	63	66	70	73	76	79	81
6.31 Hz	25	29	33	38	43	49	54	58	62	64	69	72	75	78	80
10.00 Hz	25	28	32	37	43	48	53	58	60	63	68	71	74	77	80

Table E21. Complex modulus (Pa) - Binder recovered from Mechanical foamed-RAP-160C mixture (initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	23800000	15740000	9708000	5437000	2928000	1525000	803800	428100	230600	139800	79110	41410	20950	11260	6047
0.16 Hz	21710000	13890000	10170000	5789000	3920000	1843000	1009000	542300	294200	123200	80560	47100	27920	15530	8139
0.25 Hz	31320000	21160000	13290000	7700000	4222000	2242000	1220000	671000	367500	224500	128500	70870	34320	20560	11440
0.40 Hz	35190000	24050000	15280000	8966000	5023000	2710000	1490000	839300	463300	276600	153600	88960	48290	27730	15610
0.63 Hz	40120000	27580000	17700000	10510000	5982000	3281000	1823000	1038000	583300	350000	203800	117400	63850	36990	21140
1.00 Hz	44900000	31420000	20420000	12280000	7082000	3944000	2221000	1284000	728100	409100	256500	150000	83040	48760	28230
1.59 Hz	50440000	36050000	23500000	14330000	8360000	4721000	2692000	1580000	907600	537200	322700	189900	106700	63590	37670
2.51 Hz	57220000	40650000	26990000	16600000	9856000	5642000	3259000	1936000	1126000	655100	402900	241600	138300	83390	50150
3.98 Hz	64170000	45600000	30850000	19250000	11580000	6726000	3938000	2362000	1389000	794100	494600	303700	177400	108600	65860
6.31 Hz	71650000	51760000	35140000	22280000	13560000	7992000	4746000	2884000	1710000	957000	608900	379900	226500	140500	86460
10.00 Hz	79520000	58060000	39960000	25670000	15840000	9452000	5693000	3502000	2102000	1137000	741500	472300	287800	181400	112900

Table E22. Phase angle (º) - Binder recovered from Mechanical foamed-RAP-160C mixture (initial condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	28	30	32	35	38	41	43	46	49	47	51	55	58	62	65
0.16 Hz	16	13	32	34	45	39	41	44	46	6	17	29	56	59	63
0.25 Hz	25	27	29	32	35	38	41	43	46	44	47	51	59	58	61
0.40 Hz	24	26	29	31	34	37	40	42	45	41	44	50	53	57	60
0.63 Hz	24	26	28	31	34	37	39	41	44	42	47	49	53	56	58
1.00 Hz	27	26	28	31	33	36	39	41	43	45	44	48	51	54	57
1.59 Hz	23	25	27	30	33	36	38	40	43	39	43	47	50	53	56
2.51 Hz	22	25	27	30	32	35	38	40	42	38	44	47	49	52	55
3.98 Hz	21	24	27	29	32	35	37	40	42	37	41	45	48	51	54
6.31 Hz	22	24	27	29	32	34	37	39	42	35	40	44	48	51	53
10.00 Hz	22	24	26	29	32	35	37	39	41	34	39	43	47	50	53

Table E23. Complex modulus (Pa) - Binder recovered from Mechanical foamed-RAP-160C mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	35820000	26850000	19560000	12210000	6915000	3885000	2152000	1184000	653700	277400	149100	79400	42600	21440	11890
0.16 Hz	40410000	30760000	21890000	13920000	7979000	4547000	2555000	1424000	798700	270100	163700	92140	55430	28430	15950
0.25 Hz	47700000	36650000	26000000	16880000	9858000	5731000	3286000	1870000	1070000	417100	233100	132300	73350	37910	21700
0.40 Hz	50300000	38680000	27520000	17970000	10560000	6181000	3567000	2044000	1177000	491600	312200	174200	91840	50250	29420
0.63 Hz	58680000	45290000	32570000	21640000	12940000	7717000	4540000	2652000	1557000	193400	374900	214400	122300	66200	39810
1.00 Hz	64820000	50210000	36380000	24420000	14770000	8917000	5311000	3142000	1867000	718100	433200	268400	158300	86670	52470
1.59 Hz	71500000	55600000	40560000	27490000	16820000	10270000	6194000	3710000	2232000	954200	578500	358700	201900	113200	68430
2.51 Hz	78660000	61460000	45160000	30890000	19110000	11810000	7202000	4367000	2658000	1055000	673400	421600	248900	143900	91940
3.98 Hz	86440000	67840000	50250000	34640000	21650000	13530000	8348000	5123000	3156000	1261000	827900	524000	321500	186300	117800
6.31 Hz	99350000	78480000	58670000	40940000	26010000	16530000	10370000	6475000	4057000	1444000	983500	640400	404700	237600	152100
10.00 Hz	108500000	86240000	64860000	45660000	29290000	18810000	11930000	7533000	4773000	1675000	1168000	780300	504400	304000	195800

Table E24. Phase angle (º) - Binder recovered from Mechanical foamed-RAP-160C mixture (SATS condition)

Freq\Temp	5°C	10°C	15°C	20°C	25°C	30°C	35°C	40°C	45°C	50°C	55°C	60°C	65°C	70°C	75°C
0.10 Hz	25	26	27	29	32	35	38	41	44	44	45	48	54	56	60
0.16 Hz	24	25	26	29	31	34	37	40	43	19	40	49	60	56	60
0.25 Hz	23	24	26	28	30	33	36	39	41	40	45	48	52	56	59
0.40 Hz	23	24	25	27	30	33	35	38	41	59	52	45	44	54	57
0.63 Hz	22	23	25	27	29	32	34	37	40	32	41	46	50	53	55
1.00 Hz	22	23	24	26	29	31	34	36	39	35	42	45	48	52	55
1.59 Hz	21	23	24	26	28	31	33	36	38	36	42	47	47	51	54
2.51 Hz	21	22	24	25	28	30	33	35	38	34	38	42	47	50	52
3.98 Hz	21	22	23	25	27	30	32	34	37	31	36	41	45	49	51
6.31 Hz	20	21	23	24	27	29	31	34	36	29	35	40	44	48	51
10.00 Hz	20	21	22	24	26	28	31	33	36	27	33	39	43	47	50